Prepared for:

ARMSTRONG WORLD INDUSTRIES SUPERFUND SITE
Operable Unit 2
Macon, GA
Docket Number: CERCLA-04-2018-3759

FINAL FIELD SAMPLING PLAN
Revision 1

REMEDIATION INVESTIGATION / FEASIBILITY STUDY WORK PLAN OPERABLE UNIT 2

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August 2019

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Acronyms and Abbreviations

AC	Alternating Current
COPC	Constituents of Potential Concern
DC	Direct Current
DU	Decision Unit
EPA	Environmental Protection Agency
FSP	Field Sampling Plan
GPS	Global Positioning unit
HASP	Health and Safety Plan
IDW	Investigative-Derived Waste
ISM	Incremental Sampling Methodology
ITRC	Interstate Technology Regulatory Council
Landfill Area	Remote Landfill, FMNOL, explosives demolition area
MS/MSD	Matrix Spike/ Matrix Spike Duplicate
NTU	Nephelometric Turbidity Units
NWI	National Wetlands Inventory
OU2	Operable Unit - 2
PAH	Polyaromatic Hydrocarbons
PCB	Polychlorinated Biphenyls
PID	Photoionization Detector
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
RI/FS	Remedial Investigation / Feasibility Study
RSV	Refined Ecological Screening Value
RUST	Rust Environment and Infrastructure Inc.
SAIC	Science Applications International Corporation
Site	OU-2
SOPs	Standard Operating Procedures
SU	Sampling Unit
SVOC	Semi-volatile Organic Compounds
TAL	Target Analyte List
VOC	Volatile Organic Compounds
Work Plan	RI/FS Work Plan

1 INTRODUCTION

1.1 Background

This Field Sampling Plan (FSP) is submitted on behalf of the Respondents pursuant to the Administrative Settlement Agreement and Order on Consent Docket No. CERCLA-04-2018-3759, to conduct a Remedial Investigation/Feasibility Study (RI/FS) for the Armstrong World Industries Superfund Site, Operable Unit 2 (OU2) in Macon, Georgia (Site). The purpose of the FSP is to support field activities associated with the RI/FS process for the Site, specifically the work scope presented in the RI/FS Work Plan (Work Plan) (EPS, 2019a). This FSP specifies procedures that will be followed to execute the Work Plan and will be used in conjunction with the Quality Assurance Project Plan (QAPP) (EPS, 2019b) pertinent to the sampling.

1.2 Field Sampling Plan Organization

The FSP is organized as follows:

- Section 2 describes sample collection methods that will be followed while executing the work scope;
- Section 3 provides supporting protocols for sample collection including sample custody, equipment decontamination, and quality assurance/quality control; and
- Section 4 provides a list of references cited in this document.

2 FIELD PROCEDURES

2.1 Overview

The work scope outlined in the Work Plan includes sampling of various environmental media (soil, groundwater, sediment, surface water, landfill waste material) and biota (fish), delineation of site features, and receptor surveys. Field samples will be collected per the procedures outlined in this FSP. The field sampling procedures presented in this section are generally consistent with standard operation procedures (SOPs) developed by the United States Environmental Protection Agency (EPA) Region 4. Relevant SOPs are presented in the Appendix. Laboratory analytical methods are presented in the QAPP.

2.2 Soil Sampling

Soil[1] sampling will be accomplished using incremental sampling methodology (ISM) according to the procedures outlined in the Interstate Technology Regulatory Council (ITRC) guidance (ITRC, 2012). The proposed Decision Units (DUs) are shown on Figure 1[2] and target the top 6-inches of the soil column. Note that each DU is comprised of one Sampling Unit (SU) (*i.e.*, the DU and SU are one in the same). ISM requires that the total sample mass be sufficient to represent the heterogeneity of soil particles and that a sufficient number of equal-volume increments are collected in an unbiased manner from throughout the entire SU so that all particles in the unit have an equal probability of being included in the sample. To this end, each SU will be divided into 30 or more incremental sampling grids and 3 soil aliquots will be collected at random from each incremental grid to establish 3 replicate samples for each DU (each replicate sample is comprised of 1 soil aliquot from each incremental sampling grid, *i.e.*, 30+ aliquots per replicate). Each replicate sample will be at least 1 kg.

Upon commencing the ISM fieldwork, a portable global positioning unit (GPS) will be used to locate and stake grid intersections within each DU to set sampling locations (compass and tape methods may be necessary to augment the use of the GPS in areas of dense canopy). Soil collection will be accomplished using various auger methods based on site conditions according to the procedures outlined in Section 4.3 of *Soil Sampling (SESDPROC-300-R3)* developed by the EPA.

Soils occurring in flooded areas will be collected using a stainless-steel hand auger. The hand auger bucket is advanced to the target depth (6-inches below ground surface) by simultaneously pushing and turning using an attached handle. Once full, the auger bucket is removed from the ground and a portion of its contents is screened with a photoionization detector (PID) and transferred to an individual sample container.

High-ground soils or soils protruding above the surface of flood waters will be collected using a sampling device that satisfies ITRC guidance. A field test will be conducted to evaluate at least

[1] Solid substrate occurring outside of a surface water feature (*e.g.*, Rocky Creek and the ditches).
[2] Decision Unit 18 (background) is not shown on Figure 1. This DU is shown on Figure 5.

two different sampling tools. One tool being considered is the Collect-N-Go soil sampling kit or equivalent set-up (see image below). The Collect-N-Go soil sampling kit consists of a battery-powered drill with auger drill bits and handle bucket with a collection tube located in the bottom of the bucket. The auger drill is inserted into the collection tube and advanced to target depth (6 inches into the soil). As the drill advances, the bucket is pressed firmly to the soil surface so that the soil tailings flow through the collection tube and into the bucket. The soil tailings collected in the bucket will be screened with a PID and a portion of its contents transferred to a sample container.

Collect-N-Go Soil Sample Kit (image from Nasco catalog)

Once each incremental grid in a DU is sampled, the soil aliquots (each in its own sample container) will be placed in a large bag. For each SU there will be three large bags, each of which contain 30 or more smaller containers. These large bags will be sent to the laboratory for processing. The laboratory will composite, dry, sieve and subsample the material as specified in the laboratory's Standard Operating Procedure (included in the Appendix). ISM soil samples will be analyzed according to the following test regimen:

- Metals[3] by EPA Method 6010C;
- Mercury by EPA Method 1631E;
- Polycyclic Aromatic Hydrocarbons (PAHs) by EPA Method 8270C SIM;
- Perchlorates (in the Explosives Demolition Area only) by EPA Method 6850;
- Energetics (in the Explosives Demolition Area only) by EPA Method 8330B;
- Hexavalent chromium (in the Explosives Demolition Area only) by EPA Method 7196A;

[3] Aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, cobalt, copper, iron, lead, manganese, nickel, silver, thallium, vanadium and zinc. Note that metals may be naturally occurring.

- Dioxins/furans (in the Explosives Demolition Area only) by EPA Method 8290;
- Bis(2-ethylhexyl)phthalate (in the landfills only) by EPA Method 8270D; and
- PCB Aroclors and congeners by Method 8082A.

2.3 Groundwater Sampling

The “Low-Flow” method (Section 3.5 of *Groundwater Sampling (SESDPROC-301-R4)* developed by EPA) will be used to sample groundwater monitoring wells shown on Figure 2. Purging of monitoring wells will be completed using a peristaltic pump with the tubing set at the approximate mid-portion of the screened interval of the well. Standard field parameters (*i.e.*, temperature, pH, oxidation-reduction potential, dissolved oxygen, specific conductivity, and turbidity) will be measured using a water quality meter (*e.g.*, Horiba U-22 or equivalent) and recorded during purging. Groundwater samples will be collected when pH and specific conductivity have stabilized and turbidity is below 10 NTU. Stabilization is defined as follows: pH $\pm$ 0.1 units and conductivity $\pm$ 5% for three consecutive measurements (minimum of 30 minutes of purge time). The monitoring well analytical testing regimen is comprised of the following:

- Metals by EPA Method 6010C (same list as for soil);
- Mercury by EPA Method 1631E;
- Cyanide by EPA Method 335.4;
- Dieldrin by EPA Method 8081B;
- Bis(2-ethylhexyl)phthalate by EPA Method 3520C;
- PCB Aroclors and congeners by Method 8082A;
- Specific VOCs[4] by EPA Method 8260C;
- Energetics by EPA Method 8330B;
- Total organic carbon by EPA Method SM5310; and
- Nitrates by EPA Method 300.

2.4 Sediment Sampling

Sediment[5] sampling will be accomplished using ISM according to ITRC guidance. Sediment DUs are shown on Figure 3[6] and target the top 6-inches of the sediment column. Note that DU #6 consists of 5 SUs (A, B, C, D, E); the other DUs are comprised of one SU. Each SU will be divided into 30 or more incremental sampling grids and 3 sediment aliquots will be collected at random from each incremental grid to establish 3 replicate samples for each SU (each replicate sample is

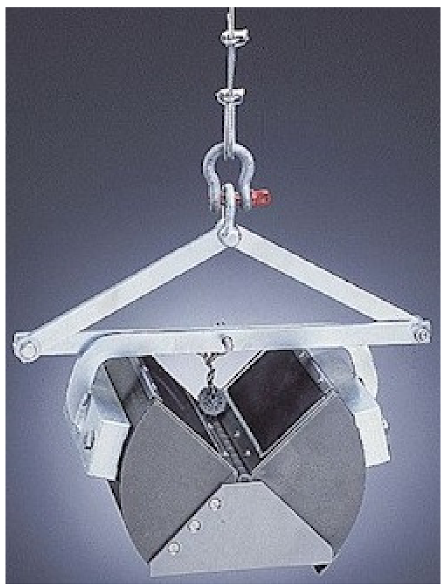
[4] 1,1-dichloroethene, carbon tetrachloride, cis-1,2-dichloroethene, tetrachloroethene, trichloroethene, and vinyl chloride

[5] Solid substrate occurring within a surface water feature (Rocky Creek and the drainage ditches).

[6] Sediment DUs 8-11 are not shown on Figure 3. These DUs are shown on Figure 5.

comprised of 1 sediment aliquot from each incremental sampling grid). Sediment collection will be performed from downstream to upstream to prevent/limit re-suspended sediment transport to downstream incremental grids.

Upon commencing the ISM fieldwork, a portable GPS will be used to locate and stake (where possible) grid intersections within each SU to set sampling locations (compass and tape methods may be necessary to augment the use of the GPS in areas of dense canopy). Sediment collection methodology will vary based on the depth of the conveyance being sampled. Dry or wadeable conveyances will be sampled with a stainless-steel scoop. If the conveyance is wadeable, sediment will be collected while facing upstream (into the current), scooping the sample along the bottom of the surface water feature in the upstream direction. In surface water bodies that are too deep to wade (such as Rocky Creek), a Ponar dredge will be used to collect sediment samples (see image below).



Ponar dredge (image from Cole-Palmer Instruments, LLC catalog)

The Ponar dredge is deployed in its open configuration from the sampling platform (boat) and lowered by rope gently to the substrate below the platform. When the Ponar strikes the bottom, the tapered cutting edges penetrate the substrate, and the rope is tugged upward closing the dredge and capturing the sample. The Ponar is then hauled to the surface where it is opened to acquire the sample. Sediment aliquots will be transferred to a sample container and all aliquots for a given replicate will be placed in one large bag.

Once each incremental grid in a SU is sampled, the large bags (each containing the smaller containers of 30+ aliquots) will be sent to the laboratory for process and subsampling. ISM sediment samples will be analyzed according to the following test regimen:

- Metals by EPA Method 6010C (same list as for soil);
- PAHs by EPA Method 82707C SIM; and
- PCB Aroclors and congeners by EPA Method 8082A.

2.5 Surface Water Sampling

Surface water sampling will be collected at the locations shown on Figure 4. Surface water samples will be collected according to Section 4 of *Surface Water Sampling (SESDPROC-201-R4)* developed by EPA using a 1-liter amber glass container. The amber will be carefully submerged 4-6 inches (where possible) below the water surface with the cap left secure on the bottle. The sampler will face upstream (into the current) when collecting the sample to prevent/limit any re-suspended sediments from entering the amber. Once the neck of the amber is submerged, the cap will be removed to fill the bottle, re-secured, and its contents transferred to the appropriate sample containers. Care will be taken not to flush any preservative out of sample containers during the fill process. The surface water analytical testing regimen will consist of:

- Metals by EPA Method 6010C (same list as for soil);
- PCB Aroclors and congeners by EPA Method 8082A; and
- Specific VOCs by EPA Method 8260C.

2.6 Seeps

Two seeps have been observed in the Landfill Area. We will field verify seep locations prior to conducting sampling and attempt to determine the amount of flow relative to recent precipitation in order to determine if it is indeed an active seep. The goal will be to sample two active seep locations per landfill or Explosives Demo Area, if they are observed. If more than two active seeps at a landfill are observed, the ones that are sampled will be based on location (to be most representative of potential landfill impact), distance from other active seeps, and rate of flow (preference for seeps that have greater flow). Seep samples adjacent to the two landfills will be analyzed for constituents being analyzed in surface water, PAHs and BEHP. Seeps adjoining the Explosives Demo Area will be analyzed for constituents being analyzed in surface water, PAHs, perchlorates and energetics.

2.7 Fish Tissue Sampling

2.7.1 Overview

Fish tissue collection is proposed at locations shown on Figure 5. Human edible game species targeted for sampling include Large-Mouth Bass (*Micropterus Salmoides*), Bluegill (*Lepomis Macrochirus*), Channel Catfish (*Ictalurus Punctatus*), Black and White Crappie (*Promoxis nigromaculatus* and *Promoxis Annularis*, respectively), and Spotted Sucker (*Minytrema Melanops*). Secondary species include Redfin Pickerel (*Esox Americanus Americanus*), Chain Pickerel (*Esox Niger*), Brown Bullhead (*Ameriurus Nebulosus*), and Redbreast Sunfish (*Lepomis Auritus*). Smaller species (or smaller size class of games species listed above) will be targeted for assessing ecological risk. Fish tissue samples from the fish collected for human exposure will be analyzed for total mercury (EPA Method 1631E) and PCB Aroclors and congeners (EPA Method 8082A). The smaller fish (4-6" in length) for ecological receptors will be frozen and analyzed in the future, if needed based on the results of the soil, sediment and surface water sampling and the progression of the ecological risk evaluation.

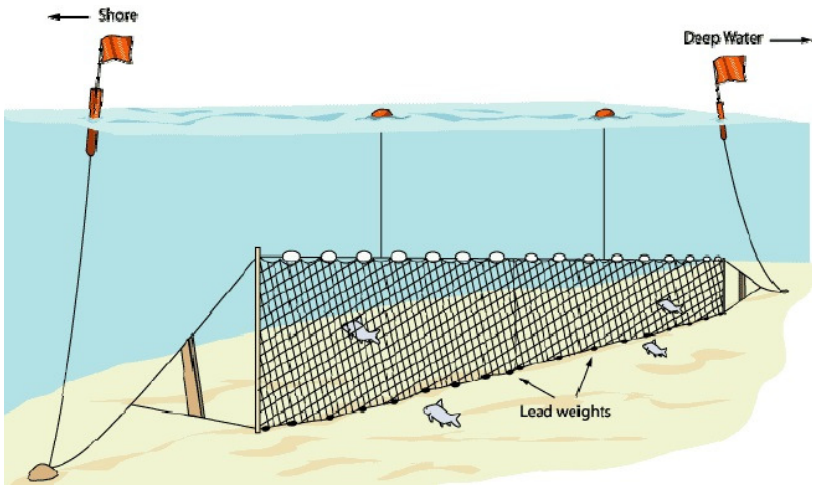
2.7.2 Collection Methods

Fish collection will be performed according to Section 3.1 (electrofishing), Section 4.2.1 (gill nets), and Section 4.3.2 (trapping), of *Fish Field Sampling (SESDPROC-512-R4)* developed by EPA. These methods are described below.

Electrofishing. Electrofishing is a form of active fish collection with the use of electricity. An electric shock is administered to immobilize the fish, which are then captured using a block net. Conditions are ideal for electrofishing when water conductivity is high (but lower than the conductivity of the fish tissue), water temperature is warm, and turbidity is low. In smaller wadeable water bodies, electrofishing may be performed using a backpack electrofisher. The backpack electrofisher set-up consists of a 24-volt battery, the backpack unit (the control box for DC current power only), a-held-held anode pole, and cathode cable hanging off the rear of the backpack unit. A minimum of two people is required to operate a backpack electrofisher: one person wears and operates the backpack electrofisher, and the other nets the fish and monitors safety. Fish collection using a backpack electrofisher is conducting moving upstream (into the current) to prevent/limit re-suspended sediments from clouding/obstructing the netter's line of sight to the fish. When a water body is large or too deep to wade, electrofishing may be performed from a boat. This set-up generally consists of: (i) a gas-powered AC generator (power source), (ii) a control box that allows for manipulation of current (AC vs DC), output voltage and pulsing, (iii) an anode attached to bow of the boat, and (iv) a cathode usually the boats hull or cable hanging over the side. A minimum of three people are required to electrofish from a boat: one person to operate the boat and monitor safety and two people to actively electrofish.

As target species are caught, they are transferred into a sample cooler with ice. Specimens not needed for analysis are released on site.

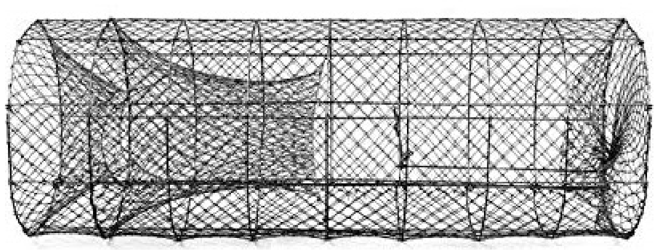
Gill Nets. A gill net consists of a single wall of mono-filament nylon mesh netting vertically suspended between a float line on top and a weighed lead line at the bottom (see below). Anchors are attached to both ends of the lead line, and surface marker buoys are attached to both ends of the float lines.



Gill net configuration (Figure 5 from Das et al, 2007)

Fish are captured in a gill net when they swim into the mesh and their bodies and gill coverings (opercles) become entangled. Gill nets will measure 25 to 50 ft. in length by 4 ft in width and consist of 2-inch or 1-1/4-inch mesh (it is anticipated that these size meshes will be optimal for collection of target species). Gill nets will be deployed and retrieved by boat. Setting the net is begun by casting the net anchor of the shoreward end of the net against the shoreline, then slowly backing the boat away from shore in the desired direction until the net is pulled taught. Once the net is taught, the offshore lead line anchor is dropped overboard causing the offshore end of the net to sink to the bottom. At the end of the desired sampling period (minimum of 4 hours), the buoy and anchor at the offshore end of the net is retrieved and the gill nets are hauled on board as the boat moves closer toward the shore. As fish are encountered in the net, they are removed by lifting the mesh over their opercula and sliding it off their bodies. If the mesh is extremely tight around the fish's body, a pick or knife may be used to free the specimen. Captured target specimens are transferred into a sample cooler and preserved with ice until later processing. Specimens not needed for analysis are released on site. The net is reset in the original manner if sampling continues at the same collection site.

Trapping. Traps are small, portable, and rigid contraptions with an opening that allows for the target species (generally bottom dwelling fish) to enter (see below). Traps are typically connected to a surface float and baited. Hoop net traps will be used to target catfish (see below).



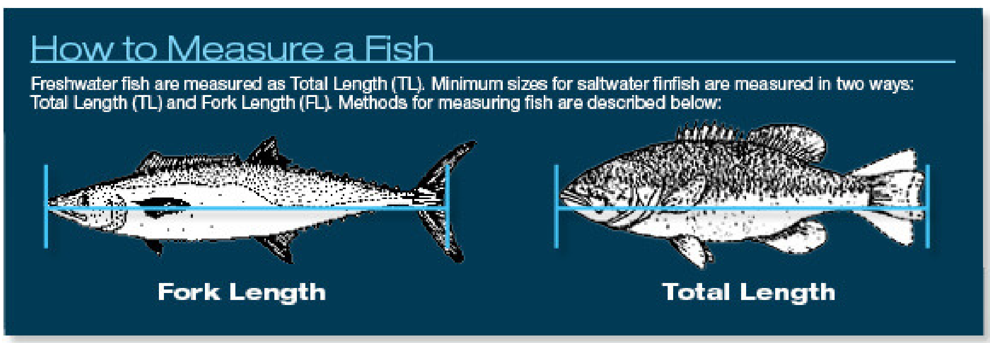
Hoop net (image from Amazon catalog)

Hoop nets will consist of 5, 30-inch openings and 1-1/4-inch mesh, and measure 7 ft in length. The hoop net traps will be deployed and retrieved by boat and simultaneously to gill nets. Captured target specimens are transferred into a sample cooler and preserved with ice until later processing.

Specimens not needed for analysis are released on site. The hoop net trap is reset in the original manner if sampling continues at the same collection site.

2.7.3 Biological Measurements and Tissue Processing

Length and weight measurements will be made on collected fish species within 24 hours after collection. Georgia Department of Natural Resources has established minimum size requirements for only one of the species targeted for collection: the Largemouth Bass at 12-inches in total length. Fish will be placed on a measuring board with the most anterior part of the fish touching the board and its side resting align the ruler of the board, and total length or fork length will be measured to the nearest 5 mm (see diagram below). Total length is measured from the most forward point of the head, with the mouth closed, to the farthest tip of the tail with the tail compressed. Fork length is measured from the most forward point of the head, with the mouth closed, to the end of the middle caudal fin rays. Once measured, the weight of the fish will be measured by analytical balance and recorded to the nearest gram.



Immediately after length and weight measurements, larger game species will be processed (i.e., scaled and fileted). Smaller species or fish will not be processed. These procedures reflect the same processes employed by fishermen to remove edible muscle tissue from fishes for consumption. Scaling and filleting will proceed as follows:

1. Cover the fillet board with a piece of aluminum foil, dull side facing up.
2. Place each fish on its side on the fillet board.
3. Remove all scales from the caudal fin to the head on both sides of the fish.
4. Make a diagonal cut from the base of the cranium, following just behind the gill to the ventral side just behind the pectoral fin.
5. Remove the flesh, including some of the belly flap, from each side of the fish by cutting from the cranium along the spine and dorsal fin to the caudal fin.
6. Place the two fillets from each fish in a labeled Ziploc® bag and freeze at -20°C.

The collection goal is three composite samples of each of the 5-target species (see Section 2.7.1) comprised of 5 specimens in each composite adhering to the "75% rule" (requires the smallest fish in the composite to be at least 75% of the length of the largest fish). This collection goal stems from the State of Georgia Department of Natural Resources fish sampling protocols. This idealistic collection goal is not typically obtainable in practice. The collected specimens will be processed, measured and archived and we will interact with agency biologists as necessary to determine which to include for analysis. For smaller species or fish, each composite will consist of 5-10 whole individuals to provide a sufficient sample mass for analysis.

2.8 Physical Assessments

2.8.1 Landfill Material Collection

Bulk samples of landfill material will be collected to estimate settlement attributable to biodegradation and dissipation of pore water from void spaces. A stainless-steel hand shovel will be used to penetrate the soil cover and to collect a sample of the underlying waste material. The bulk material will be tested for degradable organic matter (EPA Method 160.4) and moisture content (EPA Method 160.3). Soil cover materials will be stockpiled on plastic sheeting set adjacent to the work area and used to restore the site to pre-sampling conditions after sampling is complete.

2.8.2 Physical Surveys

A physical survey of surface conveyances will be performed as part of the Work Plan. Surface conveyances will be traversed from their point of origin on the Allied Industrial Park property to Rocky Creek and mapped using a survey grade GPS device for accurate depiction at scale.

2.9 Receptor Surveys

2.9.1 Well Survey

A well survey will be conducted to identify or update potential ground water use in the area. The well survey will include a desktop survey of municipal and private wells located within a three-mile radius of the Site and a windshield survey of all residences and businesses located within a 1-mile radius of the Site. Information gathered from this survey will include water usage, well construction, and well yield.

2.9.2 Surface Water Survey

A survey of Rocky Creek will be conducted from Houston Road to the Ocmulgee River to compile information pertaining to Rocky Creek's usage in the area of the Site. The survey will consist of contacting state and local agencies and field assessment. Information gathered from this survey will include locations of surface water intakes and discharges and amount of angling activity.

2.9.3 Wetlands Survey

Wetlands will be located/delineated from Houston Road to the Ocmulgee River for accurate depiction at scale. The wetlands survey will consist of a desktop analysis of United States Fish and

Wildlife's National Wetlands Inventory (NWI) and historical aerial photography followed by spot checking by field verification for the areas on the Site.

3 SUPPORTING PROTOCOLS

3.1 Sample Handling and Storage

Environmental samples collected for laboratory analysis will be placed in the containers specified in the QAPP and individual fish composites will be sealed in Zip-loc® or other similar plastic bags. Disposable gloves will be worn during sampling and discarded after each sample is collected to minimize the potential for cross-contamination. Field samples will be labeled with the Julian date and a unique identification number indicating environmental media (soil, groundwater, sediment, surface water, bulk waste) or biota (sample species and sequence number) and location/site where the sample was collected. Samples will be preserved at 4°C or frozen according to the QAPP until prepared for shipment to the analytical laboratory.

3.2 Sample Shipping and Custody

Sample labeling, shipping, and chain of custody will be performed in accordance with the procedures specified in Section 3 of *Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209-R3)* and Section 3 of *Sample and Evidence Management (SESDPROC-005-R2)* developed by the EPA. Samples will be packed for shipping in ice chests and coolers. Field samples will be shipped on wet ice to maintain a 4°C condition or dry ice to maintain frozen condition according to applicable temperature preservation requirements specified in the QAPP. Chain of custody records will be sealed in a waterproof plastic bag and taped inside the cooler lids. After packing, two custody seals will be signed, dated and affixed from the cooler lid to the cooler body to ensure that any tampering with the cooler contents would be immediately evident to sample custodians on the receiving end of the shipment. The cooler lids will be sealed with strapping tape aligned overtop of the custody seals, and the coolers will be shipped overnight to the analytical laboratory.

3.3 Logbooks

Sampling personnel will use a bound field log book with moisture-resistant pages to record pertinent sampling information with an indelible pen or marker. Logbooks will be maintained according to the SOP developed by the EPA (*Logbooks (SESDPROC-010-R5)*). Each log book will identify the project name, project number, and geographic location of the site. Daily field activities and sampling information will be entered in the log book on serially-numbered pages. At the end of each day's entries, sample-collection personnel will sign and date the entry. Corrections will be made to entries by initialed and dated line-out deletions. A diagonal line will be drawn across the remaining blank space of the last page of each day's entry.

3.4 Equipment Calibration

The following equipment will be calibrated at a daily frequency to ensure collection of accurate measurements: PID, water quality meter, and analytical balance. Calibration will be performed according to manufacturer specifications. Field personnel will maintain a logbook of equipment calibration records.

3.5 Health and Safety

All Site activities associated with this FSP will follow the procedures outline in the Site-specific Health and Safety Plan (EPS, 2019c).

3.6 Equipment Decontamination

Field equipment used to collect samples for laboratory analysis will require decontamination between samples to assure sample integrity, except in instances where the equipment is dedicated to collecting a single sample. Decontamination will be performed according to *Field Equipment Cleaning and Decontamination (SESDPROC-205-R3)* developed by the EPA. In general, sampling equipment will be decontaminated according to the following procedure:

1. Rinse with tap water and scrub with brush to remove particulate matter and surface films.
2. Wash with phosphate-free detergent (*e.g.*, Liquinox).
3. Rinse with organic-free water.
4. Allow to air dry.
5. Wrap dry equipment in aluminum foil or bag in clean plastic.

Field equipment requiring decontamination are described below categorized by environmental media.

Soil and Sediment (ISM Sampling). Equipment used to collect soil and sediment samples include hand augers, auger drill bits, Collect-N-Go bucket, homogenization container, stainless-steel spoons/scoops, and Ponar Dredge Sampler. Clean (decontaminated) equipment will be used to collect 3 replicate ISM samples per SU. Accordingly, 3 sets of decontaminated sampling equipment will be used per SU. Note that soil sampling equipment do not require decontamination between collection of soil aliquots as the aliquots are physically combined to establish an ISM sample.

Groundwater. Water level meters will be decontaminated between each well. Tubing will be dedicated to a single well and therefore not require decontamination.

Surface Water. A clean amber will be used to collect surface water at each sampling location; thus, no decontamination is required for surface water sampling.

Fish Tissue. Scaling and fileting equipment (scaler, filleting knife, fillet board, etc.) will be decontaminated between fish. Scaling and fileting equipment will be rinsed with isopropanol between Steps 3 and 4 listed above.

3.7 Quality Assurance/Quality Control

Quality Assurance/quality control (QA/QC) samples will include field duplicates, field blanks, equipment blanks, and matrix spikes/matrix spike duplicates (MS/MSDs):

- Field duplicate- A co-located sample for assessing the precision of the sampling process and integrity of the analytical result.
- Field blank- A sample prepared in the field to evaluate the potential for contamination of a sample by site constituents of potential concern (COPCs) from a source not associated with the sample collected (e.g., airborne dust or organic vapors).
- Equipment blank- A sample collected in the field to verify the efficiency of decontamination procedures.
- MS/MSD- An aliquot of a sample spiked with known concentrations of the COPC used to establish the applicability of the analytical approach to the sample matrix of interest. Comparison of the MS to the MSD will yield a precision measurement in a given matrix.

Details regarding the collection and analysis of these samples, as well as procedures for evaluation of QA/QC data, are presented in the QAPP. The frequency at QA/QC samples are analyzed generally will follow Sections 3.3.3 and 3.5 (organics) or 3.6 (inorganics) of *Field Sampling Quality Control (SESDPROC-011-R5)*. Field duplicates and MS/MSDs will be analyzed at a frequency of 5% or more frequently if required by the analytical laboratory. Field and equipment blanks will be collected at a weekly frequency.

3.8 Investigation-Derived Waste Handling

Investigation-derived wastes (IDWs) may include soil cuttings, debris (*i.e.*, concrete, stone, etc.), and wastewater (*i.e.*, decontamination fluids and purge water). These waste streams will be segregated and containerized at the point of generation according to the packaging requirements of the Department of Transportation. The IDW will be sampled as necessary to generate a waste profile and shipped off-site to the appropriate facility for treatment/disposal.

4 REFERENCES

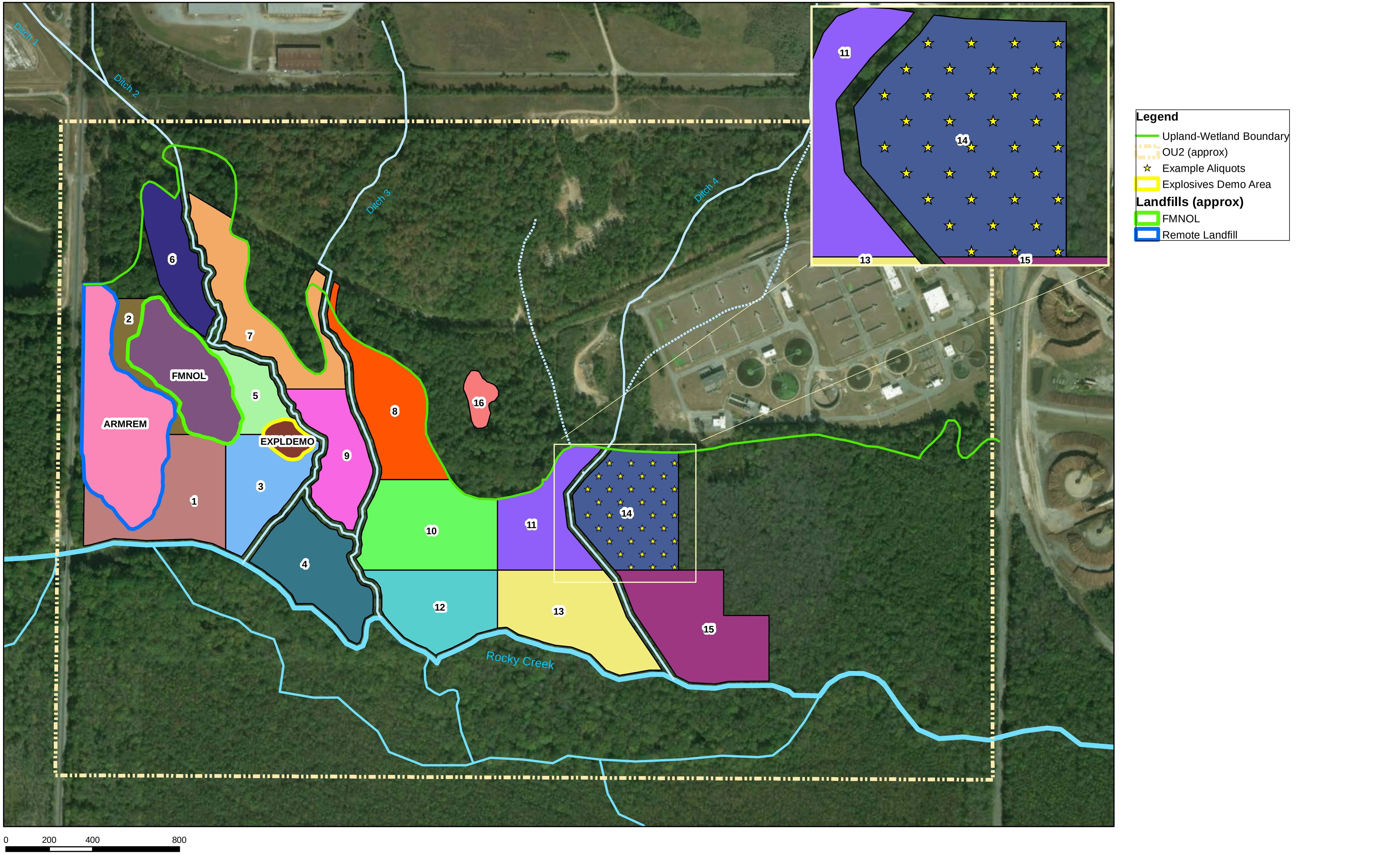
ITRC, 2012. *Incremental Sampling Methodology. ISM-1*. Washington, D.C.: Interstate Technology & Regulatory Council, Incremental Sampling Methodology Team. www.itrcweb.org.

EPS, 2019a. Remedial Investigation/Feasibility Study Work Plan, Armstrong World Industries, Operable Unit 2 (Revision 0). April 2019.

EPS, 2019b. *Quality Assurance Project Plan for Remedial Investigation/Feasibility Study, Armstrong World Industries, Operable Unit 2 (Revision 0).* April 2019.

EPS, 2019c. Health and Safety Plan for Remedial Investigation/Feasibility Study, Armstrong World Industries, Operable Unit 2 (Revision 0). April 2019

FIGURES



Soil ISM Decision Units

Figure No. 1

EPS

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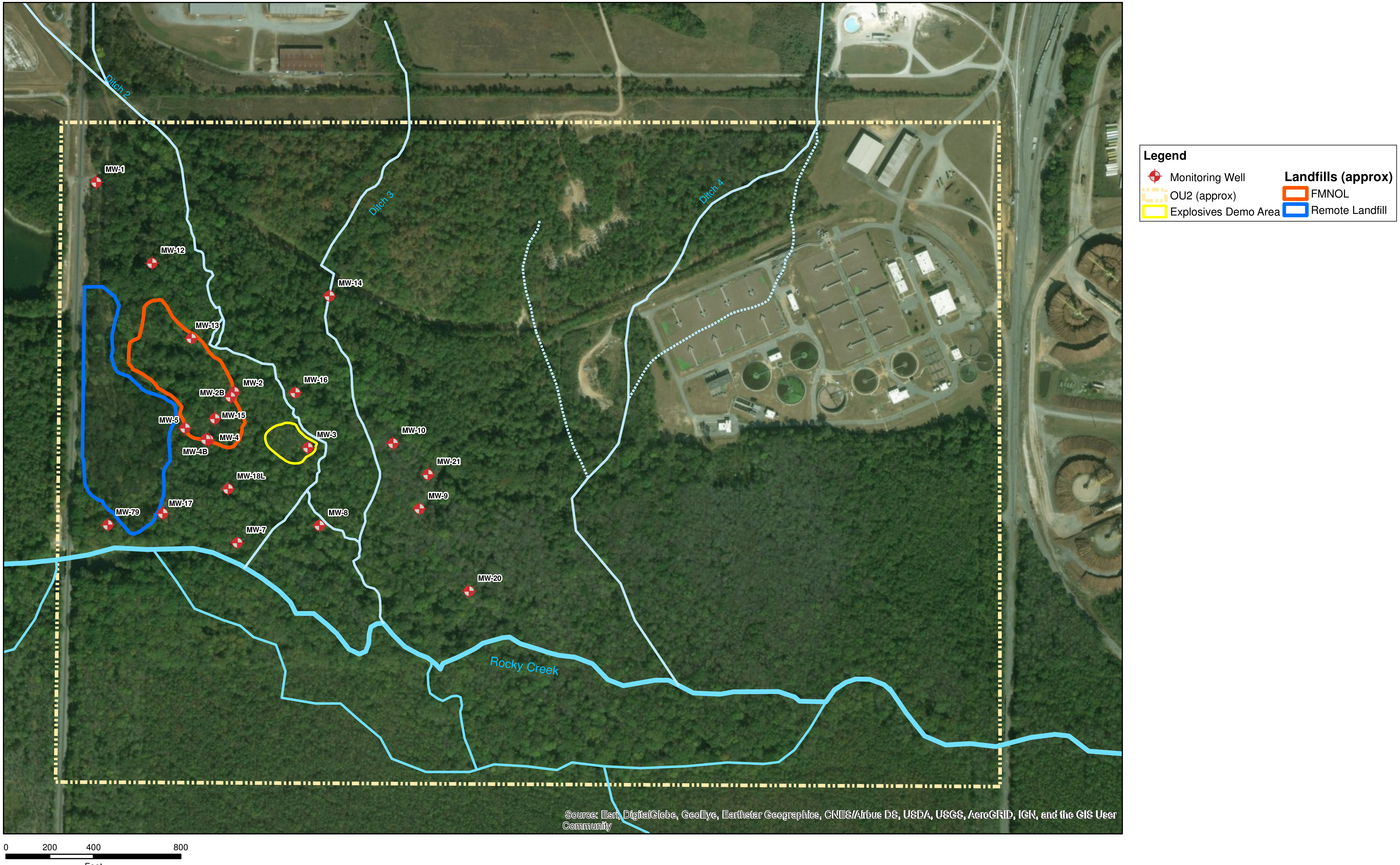

Proposed Monitoring Well Sampling

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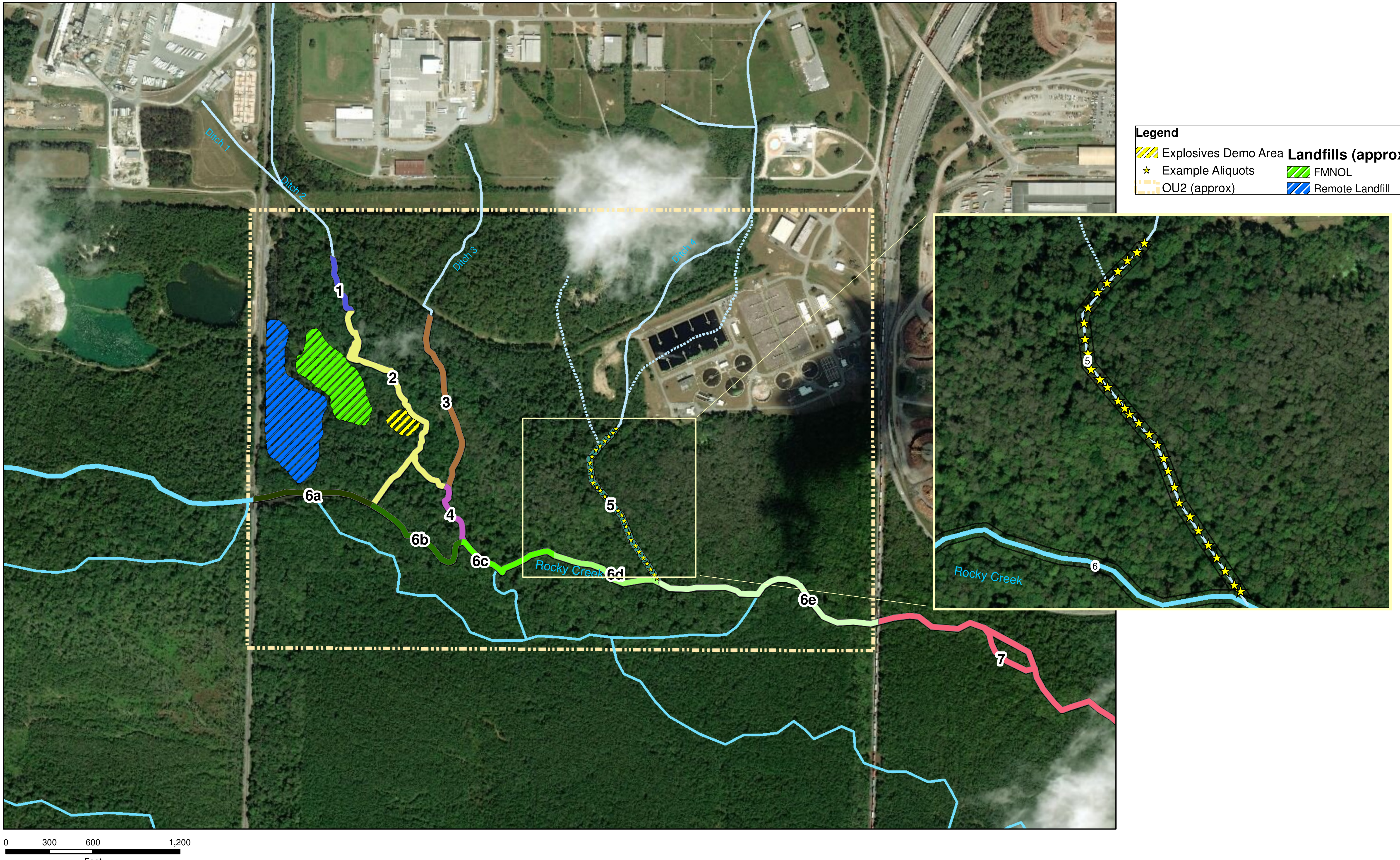
Figure No. 2



Sediment ISM Decision Units

Figure No. 3

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Proposed Surface Water Sampling Locations

Figure No. 4

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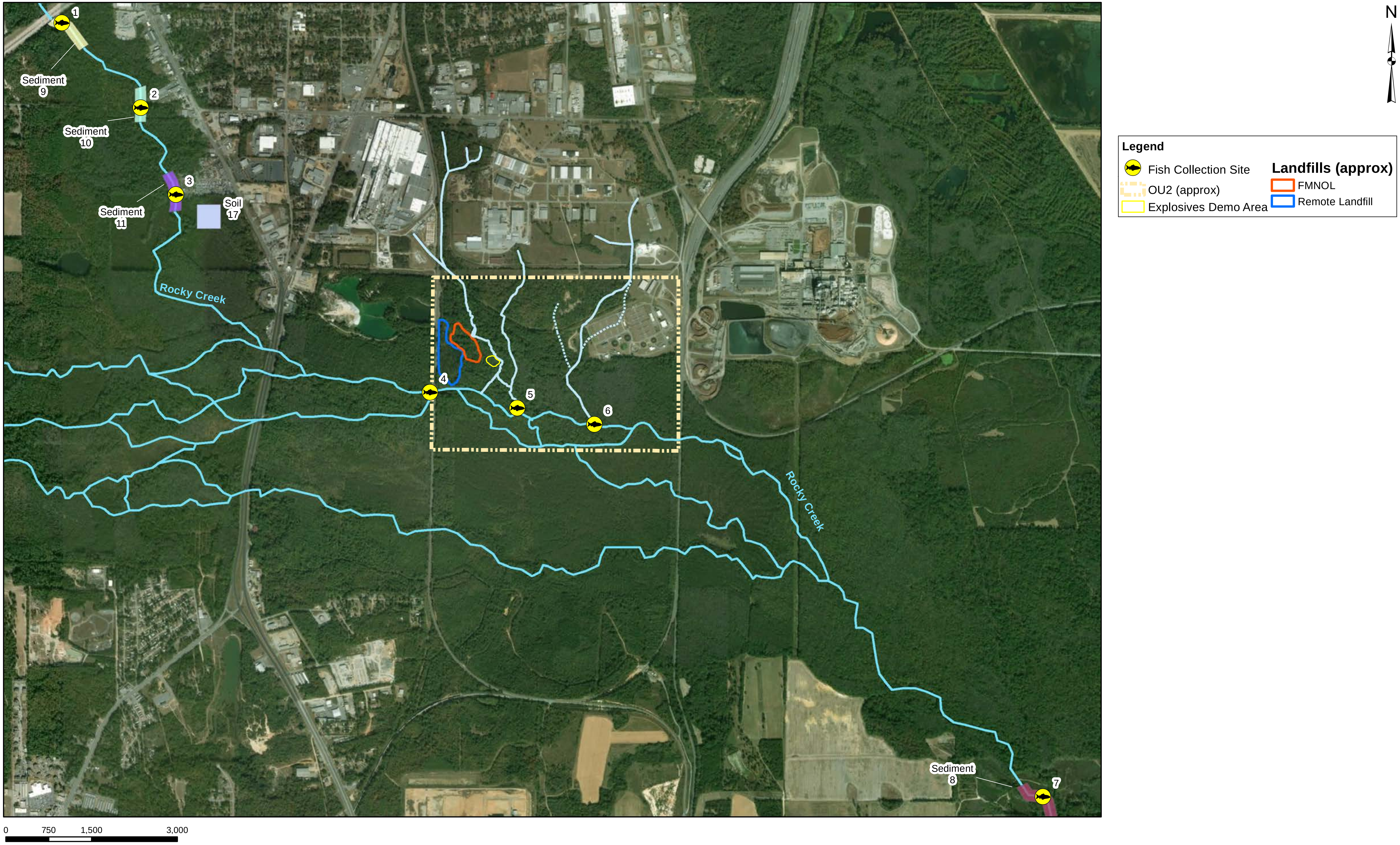

Proposed Fish Collection Sites

Figure No. 5

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F:\AWI_Macon\Macon OU2\GIS\RIFS FSP\Fig 5_Fish Sampling Locations

APPENDIX

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Region 4 U.S. Environmental Protection Agency Science and Ecosystem Support Division Athens, Georgia	
OPERATING PROCEDURE	
Title: **Soil Sampling**	
Effective Date: August 21, 2014	**Number:** SESDPROC-300-R3
Authors	
Name: Kevin Simmons Title: Environmental Scientist, Regional Expert Signature: Date: 8/18/2014	
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-300-R3, *Soil Sampling*, replaces SESDPROC-300-R2. **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Updated the author from Fred Sloan to Kevin Simmons. Updated the Enforcement and Investigations Branch Chief from Archie Lee to Acting Chief, John Deatrick. Section 1.5.1: Added "The reader should" to last sentence of the paragraph. Section 1.5.2: Omitted "When sampling in landscaped areas," from first sentence of eighth bullet. Section 3.2.4: In the first paragraph, first sentence, added "(rapidly form bubbles)." Omitted "(rapidly form bubbles)" from second paragraph, second sentence. Any reference to "Percent Moisture and Preservation Compatibility (MOICA)" or "Percent Moisture" was changed to "Percent Solids", both in the text and in Table 1.	August 21, 2014
SESDPROC-300-R2, *Soil Sampling*, replaces SESDPROC-300-R1.	December 20, 2011
SESDPROC-300-R1, *Soil Sampling*, replaces SESDPROC-300-R0.	November 1, 2007
SESDPROC-300-R0, Soil Sampling, Original Issue	February 05, 2007

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TABLES

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting soil samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling soil samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a soil sample, the variant procedure will be documented in the field logbook and subsequent investigation report, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN, and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field X-Ray Fluorescence (XRF) Measurement, SESDPROC-107, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

US EPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, Most Recent Version (Method 5035)

US EPA. Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting soil samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. The reader should address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting soil samples:

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association

(IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.

- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader in the project files.
- Sampling in landscaped areas: Cuttings should be placed on plastic sheeting and returned to the borehole upon completion of the sample collection. Any 'turf plug' generated during the sampling process should be returned to the borehole.
- Sampling in non-landscaped areas: Return any unused sample material back to the auger, drill or push hole from which the sample was collected.

2 Special Sampling Considerations

2.1 Special Precautions for Trace Contaminant Soil Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different sample is collected and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers with samples suspected of containing high concentrations of contaminants shall be handled and stored separately.
- All background samples shall be segregated from obvious high-concentration or waste samples. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area. Samples of waste or highly-contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other member(s) collect the samples.
- Samplers must use new, verified/certified-clean disposable or non-disposable equipment cleaned according to procedures contained in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205), for collection of samples for trace metals or organic compound analyses.

2.2 Sample Homogenization

1. If sub-sampling of the primary sample is to be performed in the laboratory, transfer the entire primary sample directly into an appropriate, labeled sample container(s). Proceed to step 4.

2. If sub-sampling the primary sample in the field or compositing multiple primary samples in the field, place the sample into a glass or stainless steel homogenization container and mix thoroughly. Each aliquot of a composite sample should be of the same approximate volume.

3. All soil samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. ***Samples for VOC analysis are not homogenized.*** The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
- Two quarters should then be mixed to form halves.
- The two halves should be mixed to form a homogenous matrix.

  This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4. Place the sample into an appropriate, labeled container(s) by using the alternate shoveling method and secure the cap(s) tightly. The alternate shoveling method involves placing a spoonful of soil in each container in sequence and repeating until the containers are full or the sample volume has been exhausted. Threads on the container and lid should be cleaned to ensure a tight seal when closed.

2.3 Dressing Soil Surfaces

Any time a vertical or near vertical surface is sampled, such as achieved when shovels or similar devices are used for subsurface sampling, the surface should be dressed (scraped) to remove smeared soil. This is necessary to minimize the effects of contaminant migration interferences due to smearing of material from other levels.

2.4 Quality Control

If possible, a control sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. This control sample should be collected as close to the sampled area as possible and from the same soil type. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by sampling tools. SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011) contains other procedures that may be applicable to soil sampling investigations.

2.5 Records

Field notes, recorded in a bound field logbook, as well as chain-of-custody documentation will be generated as described in the SESD Operating Procedure for Logbooks (SESDPROC-010) and the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

3 Method 5035

The procedures outlined here are summarized from *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, Method 5035.*

3.1 Soil Samples for Volatile Organic Compounds (VOC) Analysis

If samples are to be analyzed for VOCs, they should be collected in a manner that minimizes disturbance of the sample. For example, when sampling with an auger bucket, the sample for VOC analysis should be collected directly from the auger bucket (preferred) or from minimally disturbed material immediately after an auger bucket is emptied into the pan. The sample shall be containerized by filling an En Core® Sampler or other Method 5035 compatible container. ***Samples for VOC analysis are not homogenized.*** Preservatives may be required for some samples with certain variations of Method 5035. Consult the method or the principal analytical chemist to determine if preservatives are necessary.

3.2 Soil Sampling (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of VOCs in soils at a project site. Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

3.2.1 Equipment

Soil for VOC analyses may be retrieved using any of the SESD soil sampling methods described in Sections 4 through 8 of this procedure. Once the soil has been obtained, the En Core® Sampler, syringes, stainless steel spatula, standard 2-oz. soil VOC container, or pre-prepared 40 mL vials may be used/required for sub-sampling. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the site or sampling investigation. The various sub-sampling methods are described below.

3.2.2 Sampling Methodology - Low Concentrations (<200 µg/kg)

When the total VOC concentration in the soil is expected to be less than 200 µg/kg, the samples may be collected directly with the En Core® Sampler or syringe. If using the syringes, the sample must be placed in the sample container (40 mL pre-prepared vial) immediately to reduce volatilization losses. The 40 mL vials should contain 10 mL of organic-free water for an un-preserved sample or approximately

10 mL of organic-free water and a preservative. It is recommended that the 40 mL vials be prepared and weighed by the laboratory (commercial sources are available which supply preserved and tared vials). When sampling directly with the En Core® Sampler, the vial must be immediately capped and locked.

A soil sample for VOC analysis may also be collected with conventional sampling equipment. A sample collected in this fashion must either be placed in the final sample container (En Core® Sampler or 40 mL pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2-oz. soil jar) is used, the sample must be transferred to the final sample container (En Core® Sampler or 40 mL pre-prepared vial) as soon as possible, not to exceed 30 minutes.

NOTE: After collection of the sample into either the En Core® Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Soil samples may be prepared for shipping and analysis as follows:

En Core® Sampler - the sample shall be capped, locked, and secured in the original foil bag. All foil bags containing En Core® samplers are then placed in a plastic bag and sealed with custody tape, if required.

Syringe - Add about 3.7 cc (approximately 5 grams) of sample material to 40-mL pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag. Note: When using the syringes, it is important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample.

Stainless Steel Laboratory Spatulas - Add between 4.5 and 5.5 grams (approximate) of sample material to 40 mL containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag.

3.2.3 Sampling Methodology - High Concentrations (>200 µg/kg)

Based upon the data quality objectives and the detection level requirements, this high level method may also be used. Specifically, the sample may be packed into a single 2-oz. glass container with a screw cap and septum seal. The sample container must be filled quickly and completely to eliminate head space. Soils\sediments containing high total VOC concentrations may also be collected as described in Section 3.2.2, Sampling Methodology - Low Concentrations, and preserved using 10 mL methanol.

3.2.4 Special Techniques and Considerations for Method 5035

Effervescence

If low concentration samples effervesce (rapidly form bubbles) from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved, as needed, or all samples must be collected unpreserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic free water. An appropriate amount of organic free water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note: the amount of organic free water placed into the vials will have to be accurately measured.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size and the lack of sample mixing, sample representativeness for VOCs may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Sample holding times are specified in the Analytical Support Branch *Laboratory Operations and Quality Assurance Manual* (ASBLOQAM), Most Recent Version. Field investigators should note that the holding time for an un-preserved VOC soil/sediment sample on ice is 48 hours. Arrangements should be made to ship the soil/sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and/or analyze the sample within 48 hours of collection.

Percent Solids

Samplers must ensure that the laboratory has sufficient material to determine percent solids in the VOC soil/sediment sample to correct the analytical results to dry weight. If other analyses requiring percent solids determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent solids determination will be required. The sample collected for percent solids may also be used by the laboratory to check for preservative compatibility.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should be stored in a cooler with ice at all times.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities.

The summary table on the following page lists the options available for compliance with SW846 Method 5035. The advantages and disadvantages are noted for each option. SESD’s goal is to minimize the use of hazardous material (methanol and sodium bisulfate) and minimize the generation of hazardous waste during sample collection.

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Table 1: Method 5035 Summary

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect two 40 mL vials with ≈ 5 grams of sample, and one 2 oz. glass jar w/septum lid for screening, % moisture and preservative compatibility.	Screening conducted by lab.	Presently a 48-hour holding time for unpreserved samples. Sample containers must be tared.
2	Collect three En Core® samplers, and one 2 oz. glass jar w/septum lid for screening, % solids.	Lab conducts all preservation/preparation procedures.	Presently a 48- hour holding time for preparation of samples.
3	Collect two 40 mL vials with 5 grams of sample and preserve w/methanol or sodium bisulfate, and one 2-oz. glass jar w/septum lid for screening, % solids .	High level VOC samples may be composited. Longer holding time.	Hazardous materials used in the field. Sample containers must be tared.
4	Collect one 2-oz. glass jar w/septum lid for analysis, % solids (high level VOC only).	Lab conducts all preservation/preparation procedures.	May have significant VOC loss.

4 Manual Soil Sampling Methods

4.1 General

These methods are used primarily to collect surface and shallow subsurface soil samples. Surface soils are generally classified as soils between the ground surface and 6 to 12 inches below ground surface. The most common interval is 0 to 6 inches; however, the data quality objectives of the investigation may dictate another interval, such as 0 to 3 inches for risk assessment purposes. The shallow subsurface interval may be considered to extend from approximately 12 inches below ground surface to a site-specific depth at which sample collection using manual collection methods becomes impractical.

If a thick, matted root zone, gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials.

When compositing, make sure that each composite location (aliquot) consist of equal volumes, i.e., same number of equal spoonfuls.

4.2 Spoons

Stainless steel spoons may be used for surface soil sampling to depths of approximately 6 inches below ground surface where conditions are generally soft and non-indurated, and there is no problematic vegetative layer to penetrate.

4.2.1 Special Considerations When Using Spoons

- When using stainless steel spoons, consideration must be given to the procedure used to collect the volatile organic compound sample. If the soil being sampled is cohesive and holds its in situ texture in the spoon, the En Core® Sampler or syringe used to collect the sub-sample for Method 5035 should be plugged directly from the spoon. If, however, the soil is not cohesive and crumbles when removed from the ground surface for sampling, consideration should be given to plugging the sample for Method 5035 directly from the ground surface at a depth appropriate for the investigation Data Quality Objectives.

4.3 Hand Augers

Hand augers may be used to advance boreholes and collect soil samples in the surface and shallow subsurface intervals. Typically, 4-inch stainless steel auger buckets with cutting heads are used. The bucket is advanced by simultaneously pushing and turning using an attached handle with extensions (if needed).

4.3.1 Surface Soil Sampling

When conducting surface soil sampling with hand augers, the auger buckets may be used with a handle alone or with a handle and extensions. The bucket is advanced to the appropriate depth and the contents are transferred to the homogenization container for processing. Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

4.3.2 Subsurface Soil Sampling

Hand augers are the most common equipment used to collect shallow subsurface soil samples. Auger holes are advanced one bucket at a time until the sample depth is achieved. When the sample depth is reached, the bucket used to advance the hole is removed and a clean bucket is attached. The clean auger bucket is then placed in the hole and filled with soil to make up the sample and removed.

The practical depth of investigation using a hand auger depends upon the soil properties and depth of investigation. In sand, augering is usually easily performed, but the depth of collection is limited to the depth at which the sand begins to flow or collapse. Hand augers may also be of limited use in tight clays or cemented sands. In these soil types, the greater the depth attempted, the more difficult it is to recover a sample due to increased friction and torqueing of the hand auger extensions. At some point these problems become so severe that power equipment must be used.

4.3.3 Special Considerations for Soil Sampling with the Hand Auger

- Because of the tendency for the auger bucket to scrape material from the sides of the auger hole while being extracted, the top several inches of soil in the auger bucket should be discarded prior to placing the bucket contents in the homogenization container for processing.
- Observe precautions for volatile organic compound (VOC) sample collection found in Section 3, Method 5035. Collect the VOC sample directly from the auger bucket, if possible.
- Power augers, such as the Little Beaver® and drill rigs may be used to advance boreholes to depths for subsurface soil sampling with the hand auger. They may not be used for sample collection. When power augers are used to advance a borehole to depth for sampling, care must be taken that exhaust fumes, gasoline and/or oil do not contaminate the borehole or area in the immediate vicinity of sampling.
- When moving to a new sampling location, the entire hand auger assembly must be replaced with a properly decontaminated hand auger assembly.

5 Direct Push Soil Sampling Methods

5.1 General

These methods are used primarily to collect shallow and deep subsurface soil samples. Three samplers are available for use within the Division's direct push tooling inventory. All of the sampling tools involve the collection and retrieval of the soil sample within a thin-walled liner. The following sections describe each of the specific sampling methods that can be accomplished using direct push techniques, along with details specific to each method. While SESD currently uses the sample tooling described, tooling of similar design and materials is acceptable.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with these devices.

5.2 Large Bore® Soil Sampler

The Large Bore® (LB) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of depth-discrete subsurface soil samples. The sample barrel is approximately 30-inches (762 mm) long and has a 1.5-inch (38 mm) outside diameter. The LB® sampler is capable of recovering a discrete sample core 22 inches x 1.0 inch (559 mm x 25 mm) contained inside a removable liner. The resultant sample volume is a maximum of 283 mL.

After the LB® sample barrel is equipped with the cutting shoe and liner, the piston-rod point assembly is inserted, along with the drive head and piston stop assembly. The assembled sampler is driven to the desired sampling depth, at which time the piston stop pin is removed, freeing the push point. The LB® sampler is then pushed into the soil a distance equal to the length of the LB® sample barrel. The probe rod string, with the LB® sampler attached, is then removed from the subsurface. After retrieval, the LB® sampler is then removed from the probe rod string. The drive head is then removed to allow removal of the liner and soil sample.

5.3 Macro-Core® Soil Sampler

The Macro-Core® (MC) sampler is a solid barrel direct push sampler equipped with a piston-rod point assembly used primarily for collection of either continuous or depth-discrete subsurface soil samples. Although other lengths are available, the standard MC® sampler has an assembled length of approximately 52 inches (1321 mm) with an outside diameter of 2.2 inches (56 mm). The MC® sampler is capable of recovering a discrete sample core 45 inches x 1.5 inches (1143 mm x 38 mm) contained inside a removable liner. The resultant sample volume is a maximum of 1300 mL. The MC® sampler may be used in either an open-tube or closed-point configuration. Although the MC® sampler can be used as an open-barrel sampler, in SESD usage, the piston point is always used to prevent the collection of slough from the borehole sides.

5.4 Dual Tube Soil Sampling System

The Dual Tube 21 soil sampling system is a direct push system for collecting continuous core samples of unconsolidated materials from within a sealed outer casing of 2.125-inch (54 mm) OD probe rod. The samples are collected within a liner that is threaded onto the leading end of a string of 1.0-inch diameter probe rod. Collected samples have a volume of up to 800 mL in the form of a 1.125-inch x 48-inch (29 mm x 1219 mm) core. Use of this method allows for collection of continuous core inside a cased hole, minimizing or preventing cross-contamination between different intervals during sample collection. The outer casing is advanced, one core length at a time, with only the inner probe rod and core being removed and replaced between samples. If the sampling zone of interest begins at some depth below ground surface, a solid drive tip must be used to drive the dual tube assembly and core to its initial sample depth.

5.5 Special Considerations When Using Direct Push Sampling Methods

- *Liner Use and Material Selection* – Direct Push Soil Samples are collected within a liner to facilitate removal of sample material from the sample barrel. The liners may only be available in a limited number of materials for a given sample tool, although overall, liners are available in brass, stainless steel, cellulose acetate butyrate (CAB), polyethylene terepthalate glycol (PETG), polyvinyl chloride (PVC) and Teflon®. For most SESD investigations, the standard polymer liner material for a sampling tool will be acceptable. When the study objectives require very low reporting levels or unusual contaminants of concern, the use of more inert liner materials such as Teflon® or stainless steel may be necessary.

- *Sample Orientation* – When the liners and associated sample are removed from the sample tubes, it is important to maintain the proper orientation of the sample. This is particularly important when multiple sample depths are collected from the same push. It is also important to maintain proper orientation to define precisely the depth at which an aliquot was collected. Maintaining proper orientation is typically accomplished using vinyl end caps. Convention is to place red caps on the top of the liner and black caps on the bottom to maintain proper sample orientation. Orientation can also be indicated by marking on the exterior of the liner with a permanent marker.

- *Core Catchers* – Occasionally the material being sampled lacks cohesiveness and is subject to crumbling and falling out of the sample liner. In cases such as these, the use of core catchers on the leading end of the sampler may help retain the sample until it is retrieved to the surface. Core catchers may only be available in specific materials and should be evaluated for suitability. However, given the limited sample contact that core-catchers have with the sample material, most standard core-catchers available for a tool system will be acceptable.

- *Decontamination* – The cutting shoe and piston rod point are to be decontaminated between each sample, using the procedures specified for the collection of trace organic and inorganic compounds found in Field Equipment and Decontamination – SESDPROC-205, most recent version. Within a borehole, the sample barrel, rods, and drive head may be subjected to an abbreviated cleaning to remove obvious and loose material, but must be cleaned between boreholes using the procedures specified for downhole drilling equipment in Field Equipment and Decontamination – SESDPROC-205, most recent version.

- *Decommissioning* – Boreholes must be decommissioned after the completion of sampling. Boreholes less than 10 feet deep that remain open and do not approach the water table may be decommissioned by pouring 30% solids bentonite grout from the surface or pouring bentonite pellets from the surface, hydrating the pellets in lifts. Boreholes deeper than 10 feet, or any borehole that intercepts groundwater, must be decommissioned by pressure grouting with 30% solids bentonite grout, either through a re-entry tool string or through tremie pipe introduced to within several feet of the borehole bottom.

- *VOC Sample Collection* – Observe precautions for volatile organic compound sample collection found in Section 3 of this procedure.

6 Split Spoon/Drill Rig Methods

6.1 General

Split spoon sampling methods are used primarily to collect shallow and deep subsurface soil samples. All split spoon samplers, regardless of size, are basically split cylindrical barrels that are threaded on each end. The leading end is held together with a beveled threaded collar that functions as a cutting shoe. The other end is held together with a threaded collar that serves as the sub used to attach the spoon to the string of drill rod. Two basic methods are available for use, including the smaller diameter standard split spoon, driven with the drill rig safety hammer, and the larger diameter continuous split spoon, advanced inside and slightly ahead of the lead auger during hollow stem auger drilling. The following sections describe each of the specific sampling methods, along with details specific to each method.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with these devices.

6.2 Standard Split Spoon

A drill rig is used to advance a borehole to the target depth. The drill string is then removed and a standard split spoon is attached to a string of drill rod. Split spoons used for soil sampling must be constructed of stainless steel and are typically 2.0-inches OD (1.5-inches ID) and 18-inches to 24-inches in length. Other diameters and lengths are common and may be used if constructed of the proper material. After the spoon is attached to the string of drill rod, it is lowered into the borehole. The safety hammer is then used to drive the split spoon into the soil at the bottom of the borehole. After the split spoon has been driven into the soil, filling the spoon, it is retrieved to the surface, where it is removed from the drill rod string and opened for sample acquisition.

6.3 Continuous Split Spoon

The continuous split spoon is a large diameter split spoon that is advanced into the soil column inside a hollow stem auger. Continuous split spoons are typically 3 to 5 inches in diameter and either 5 feet or 10 feet in length, although the 5-foot long samplers are most common. After the auger string has been advanced into the soil column a distance equal to the length of the sampler being used it is returned to the surface. The sampler is removed from inside the hollow stem auger and the threaded collars are removed. The split spoon is then opened for sampling.

6.4 Special Considerations When Using Split Spoon Sampling Methods

- Always discard the top several inches of material in the spoon before removing any portion for sampling. This material normally consists of borehole wall material that has sloughed off of the borehole wall after removal of the drill string prior to and during inserting the split spoon.
- Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

7 Shelby Tube/Thin-Walled Sampling Methods

7.1 General

Shelby tubes, also referred to generically as thin-walled push tubes or Acker thin-walled samplers, are used to collect subsurface soil samples in cohesive soils and clays during drilling activities. In addition to samples for chemical analyses, Shelby tubes are also used to collect relatively undisturbed soil samples for geotechnical analyses, such as hydraulic conductivity and permeability, to support hydrogeologic characterizations at hazardous waste and other sites.

If gravel, concrete, etc. is present at or near the surface, it should be removed before the sample is collected. The depth measurement for the sample begins at the top of the soil horizon, immediately following any removed materials. Turf grass is not typically removed prior to sampling with this device.

7.2 Shelby Tube Sampling Method

A typical Shelby tube is 30 inches in length and has a 3.0-inch OD (2.875-inch ID) and may be constructed of steel, stainless steel, galvanized steel, or brass. They also typically are attached to push heads that are constructed with a ball-check to aid in holding the contained sample during retrieval. If used for collecting samples for chemical analyses, it must be constructed of stainless steel. If used for collecting samples for standard geotechnical parameters, any material is acceptable.

To collect a sample, the tube is attached to a string of drill rod and is lowered into the borehole, where the sampler is then pressed into the undisturbed material by hydraulic force. After retrieval to the surface, the tube containing the sample is then removed from the sampler head. If samples for chemical analyses are needed, the soil contained inside the tube is then removed for sample acquisition. If the sample is collected for geotechnical parameters, the tube is typically capped, maintaining the sample in its relatively undisturbed state, and shipped to the appropriate geotechnical laboratory.

7.3 Special Considerations When Using Split Spoon Sampling Methods

Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

8 Backhoe Sampling Method

8.1 General

Backhoes may be used in the collection of surface and shallow subsurface soil samples. The trenches created by excavation with a backhoe offer the capability of collecting samples from very specific intervals and allow visual correlation with vertically and horizontally adjacent material. If possible, the sample should be collected without entering the trench. Samples may be obtained from the trench wall or they may be obtained directly from the bucket at the surface. The following sections describe various techniques for safely collecting representative soil samples with the aid of a backhoe.

The depth measurement for the sample begins at the top of the soil horizon.

8.2 Scoop-and-Bracket Method

If a sample interval is targeted from the surface, it can be sampled using a stainless steel scoop and bracket. First a scoop and bracket are affixed to a length of conduit and is lowered into the backhoe pit. The first step is to take the scoop and scrape away the soil comprising the surface of the excavated wall. This material likely represents soil that has been smeared by the backhoe bucket from adjacent material. After the smeared material has been scraped off, the original stainless steel scoop is removed and a clean stainless steel scoop is placed on the bracket. The clean scoop can then be used to remove sufficient volume of soil from the excavation wall to make up the required sample volume.

8.3 Direct-from-Bucket Method

It is also possible to collect soil samples directly from the backhoe bucket at the surface. Some precision with respect to actual depth or location may be lost with this method but if the soil to be sampled is uniquely distinguishable from the adjacent or nearby soils, it may be possible to characterize the material as to location and depth. In order to ensure representativeness, it is also advisable to dress the surface to be sampled by scraping off any smeared material that may cross-contaminate the sample.

8.4 Special Considerations When Sampling with a Backhoe

- Do not physically enter backhoe excavations to collect a sample. Use either procedure 8.2, Scoop-and-Bracket Method, or procedure 8.3, Direct-from-Bucket Method to obtain soil for sampling.
- Smearing is an important issue when sampling with a backhoe. Measures must be taken, such as dressing the surfaces to be sampled (see Section 2.3), to mitigate problems with smearing.

- Paint, grease and rust must be removed and the bucket decontaminated prior to sample collection.
- Observe precautions for volatile organic compound sample collection found in Section 3, Method 5035.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Groundwater Sampling**	
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

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SESDPROC-301-R4, Groundwater Sampling, replaces SESDPROC-301-R3. **General:** Corrected any typographical, grammatical, and/or editorial errors. **General:** An extensive rewrite and reorganization of material. Stronger support of low-flow methods while maintaining cautious view of minimal/no purge methods.	April 26, 2017
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SESDPROC-301-R0, Groundwater Sampling, Original Issue	**February 05, 2007**

TABLE OF CONTENTS

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting groundwater samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling groundwater samples in the field. On the occasion that SESD field personnel determine that any of the procedures described are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a groundwater sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

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SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Temperature Measurement, SESDPROC-102, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Groundwater Level and Well Depth Measurement, SESDPROC-105, Most Recent Version

SESD Operating Procedure for Management of Investigation Derived Waste, SESDROC-202, Most Recent Version

SESD Operating Procedure for Pump Operation, SESDPROC-203, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

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1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting groundwater samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. The reader should address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting groundwater samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Always sample from the anticipated cleanest, i.e., least contaminated location, to the most contaminated location. This minimizes the opportunity for cross-contamination to occur during sampling.
- Collected samples must remain in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done legibly, completely, and neatly in a bound logbook.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Groundwater samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either pre-preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas unpreserved samples have only a seven-day holding time. In the majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the groundwater has a high amount of dissolved limestone, i.e., is highly calcareous, there will likely be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles and rendering the sample unacceptable. In this case, unpreserved vials should be used and arrangements confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a meniscus at the top of the vial and no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm or knuckle to check if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected. While the 8260 method allows for bubbles up to 6 mm at the time of analysis, dissolved or entrained gases can coalesce during shipment. Collecting VOC vials absent of bubbles is generally feasible and is a reasonable precaution.

2.2 Special Precautions for Trace Contaminant Groundwater Sampling

- Sampling equipment must be constructed of Teflon® or stainless steel materials. Bailers and pumps should be of Teflon® and stainless steel construction throughout.
- New Teflon® tubing should be used at each well, although tubing dedicated to a particular well may be reused, either after decontamination or storage in the well between sampling events. Caution is appropriate in reusing tubing where early sampling events report high concentrations of contaminants.
- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.

- Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if purging and sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same cooler as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Clean plastic sheeting will be placed on the ground at each sample location to prevent or minimize contaminating sampling equipment by accidental contact with the ground surface.
- Samplers must use new, verified certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Groundwater samples will typically be collected from the discharge line of a pump or from a bailer. Efforts should be made to reduce the flow from either the pump discharge line or the bailer during sample collection to minimize sample agitation.

2. During sample collection, make sure that the pump discharge line or the bailer does not contact the sample container.

3. Place the sample into appropriate, labeled containers. Samples collected for VOC, and alkalinity analysis must be collected without headspace. All other sample containers must be filled with an allowance for ullage.

4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If pre-preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by Analytical Services Branch (ASB) personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples except for the samples collected for VOC analysis. If additional preservative is needed, it should be added to achieve adequate preservation. Preservation requirements for groundwater samples are found in the USEPA Region 4 Analytical Services Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), most recent version.

5. Sample containers should be placed in an ice-filled cooler as soon as possible after filling. Ice in coolers should be in bags with minimal pooled water and the cooler should be periodically checked and replenished to maintain sample storage temperature.

2.4 Quality Control

Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers, tubing, or other sampling equipment.

Where appropriate, a background sample upgradient of all known influences or a control sample upgradient of site influences may be indicated. Background and control samples should be collected as close to the sampled area as possible and from the same water-bearing formation as the site samples.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Procedure for Sample and Evidence Management, SESDPROC-005.

3 Groundwater Purging and Sampling

3.1 Overview of Purging and Sampling Strategies

Purging is the process of removing stagnant water from a well, immediately prior to sampling, causing its replacement by groundwater from the adjacent formation that is representative of aquifer conditions. Sampling is the process of obtaining, containerizing, and preserving (when required) a ground water sample after the purging process is complete. There are several approaches to well purging and sampling that may be appropriate in various circumstances or for various combinations of available equipment. They are briefly summarized below and in *Table 1, Purge and Sample Strategies with Equipment Considerations.*

The M**ultiple-Volume Purge** method involves removing a minimum of three well volumes of water from the top of the water column and then sampling when the well has achieved stability of water quality parameters and adequately low turbidity. This is a traditional method and consistent results are generally obtained with samplers of varying skill. A drawback is that large volumes of purge water may be produced for large diameter or deep wells.

The **Low-Flow** method involves purging the well at a relatively low flow rate that minimizes drawdown, with the pump or tubing inlet located within the screened interval of the well. The well is sampled when water quality parameters are stable, adequately low turbidity is achieved, and the water level has achieved a stable drawdown (an unchanging water level). This method is often faster than Multiple-Volume Purge and generates less purge water. The method requires more skill and judgment on the part of the samplers.

The **Multiple-Volume Purge** method and the **Low-Flow** method can be considered equivalent for conventionally screened and filter-packed wells in that they both sample a flow-weighted average of water entering the well during pumping. However, other variables can result in differences between results with the two methods. In repeat sampling events, the sampling design should not change from one method to the other without appropriate cause. The transition should be noted in the report.

Minimum-Purge and **No-Purge** methods are based on the assumption that water within the screened interval of the well is at equilibrium with the water in the surrounding aquifer. This assumption should be carefully considered in the use of these methods and various cautions are discussed in sections below. The minimal-purge and no-purge methods are most useful for long-term monitoring and are generally inappropriate for the early stages of investigation. In some cases the methods might be used to gather screening-level data from wells that are too large to practically purge or have other sampling complications.

The **Minimum-Purge** and **No-Purge** methods collect water in the vicinity of the device under near-static conditions and are not equivalent to the multiple-volume purge and Low-Flow methods. Stratification of horizontal flow or vertical flow conditions within the well can result in non-intuitive and deceptive results. A comparison study should be conducted before transitioning a sampling program to the minimal-purge or no-purge methods.

3.2 Purging

Wells are purged to eliminate stagnant water residing in the casing and/or screen that has undergone geochemical changes or loss of VOCs. At the conclusion of purging, the desired flow-weighted average of water entering the well under pumping conditions will be available for sampling. Turbidity is often elevated during purging by the disturbance of formation materials at the borehole walls. As many contaminants (metals and many organics) will sorb to the formation particles, a sample including these particles will not represent the dissolved concentrations of the contaminants. Thus, a secondary goal of purging is to reduce the turbidity to the point that the sample will represent the dissolved concentration of contaminants.

In order to determine when a well has been adequately purged, field investigators should monitor, at a minimum, the pH, specific conductance and turbidity of the groundwater removed and the volume of water removed during purging. The measurements should be recorded in a purge table in the field logbook that includes the start time of purging, the parameter measurements at intervals during purging, estimated pumped volumes, depths to water for Low-Flow sampling, and any notes of unusual conditions. A typical purge table used for Low-Flow sampling is reproduced below.

Continuation of sample GW 65-0713

INITIAL 23.33 ft

TIME	pH (S.U.s)	Spec. Cond. (us/cm)	Temp. (Deg. C)	D.O. (mg/L)	D.O. (% sat.)	ORP (mV)	Turbidity (NTUs)	Water Level (Ft.)	Purge Vol. (gallons)
0930								Pump On	
0935	5.71	1065	19.6	0.77	8.7	43.9	210	24.83	1/4
1004	5.64	988	20.0	0.36	3.9	222.5	17.8	25.24	2
1026	5.63	959	20.5	0.25	2.7	98	9.95	25.18	3 1/2
1038	5.62	950	20.5	0.21	2.4	75	9.85	25.18	4
1046	5.61	946	20.8	0.21	2.4	73	6.07	25.18	4 1/2
1047								Sample Collection Time	

3.3 Parameter Stabilization Criteria

With respect to the ground water chemistry, an adequate purge is achieved when the pH and specific conductance of the ground water have stabilized and the turbidity has either stabilized or is below 10 Nephelometric Turbidity Units (NTUs) (twice the Secondary Drinking Water Standard of 5 NTUs).

Stabilization occurs when, for at least three consecutive measurements, the pH remains constant within 0.1 Standard Unit (SU) and specific conductance varies no more than 5 percent. Other parameters, such as dissolved oxygen (DO) or oxidation-reduction potential (ORP), may also be used as a purge adequacy parameter. Normal stability goals for DO are 0.2 mg/L or 10% change in saturation, whichever is greater. DO and ORP measurements must be conducted using either a flow-through cell or an over-topping cell to minimize oxygenation of the sample during measurement. A reasonable ORP stability goal is a range of 20 mV, although ORP is rarely at equilibrium in environmental media and often will not demonstrate enough stability to be used as a purge stabilization parameter. Determining the frequency of measurements has generally been left to 'Best Professional Judgement'. Care is in order, as measurements recorded at frequent intervals with low flow rates can falsely indicate stability of parameters. Several measurements should be made early in the well purge to establish the direction and magnitude of trends, which can then inform the stability decision. Stability parameters should either be not trending, or approaching an asymptote, when a stability determination is made. As a matter of practice, parameter measurements are generally made at 5-10 minute intervals.

Because the measured groundwater temperature during purging is subject to changes related to surface ambient conditions and pumping rates, its usefulness is subject to question for the purpose of determining parameter stability. As such, it has been removed from SESD's list of parameters used for stability determination. Even though temperature is not used to determine stability, it is still advisable to record the temperature of purge water as it is often used in the interpretation of other parameters.

Information on conducting the stability parameter measurements is available in the SESD Operating Procedures for Field pH Measurement (SESDPROC-100), Field Specific Conductance Measurement (SESDPROC-101), Field Temperature Measurement (SESDPROC-102), Field Turbidity Measurement (SESDPROC-103), Field Measurement of Dissolved Oxygen (SESDPROC-106) and Field Measurement of Oxidation-Reduction Potential (SESDPROC-113).

Table 1, Purge and Sample Strategies with Equipment Considerations

Purging Strategy	Purge Eqpt	Sample Eqpt	Comments
Multi-Volume Purge	------------------------------	------------------------------	Overall Method Comments- Advantages: Consistent results can be achieved with minimal skill level required. Common, simple equipment can be used. Disadvantages: Can result in large volumes of purge water. Can take extended periods of time with large diameter wells or long water columns.
In this traditional method, 3-5 well volumes of water are removed from the top of the water column while verifying the stability of water quality parameters. Following the well purge, the well is sampled from the top of the water column.	Bailer	Bailer	Bailers are rarely used for purging due to the effort required, the difficulty of lowering turbidity adequately, and the possibility of aerating the upper water column.
	Electric Submersible Pump	Bailer	Common multiple-volume setup when depth to water exceeds 25 feet. Abbreviated pump decontamination procedure can be used between wells.
	Electric Submersible Pump	Electric Submersible Pump	Requires full pump decontamination and new tubing at each well. In most cases the pump would be deployed to the screened interval instead to perform Low-Flow sampling.
	Peristaltic Pump	Peristaltic Pump	Common, multi-volume setup when depth to water is less than 25 feet. Special sampling techniques are required for the collection of SVOCs and VOCs.
Low-Flow methods	------------------------------	------------------------------	Overall Method Comments- Advantages: Lower volumes of purge water. May be faster, especially with longer water columns. Disadvantages: Requires greater skill for consistent results. Higher tubing costs than multi-volume method.
The pump or tubing inlet is placed within the screened interval and the well is purged to stable water quality parameters while maintaining stable drawdown of the water level.	Electric Submersible Pump	Electric Submersible Pump	Commonly used when depth to water exceeds 25 feet. Pump is cleaned to sample equipment standards prior to sampling each well and new or dedicated tubing used for each well. Concerns have been raised concerning VOC loss from agitation in the turbine section or from sample heating.
	Peristaltic Pump	Peristaltic Pump	Commonly used where depth to water is less than 25 feet. Special sampling techniques required for the collection of SVOCs and VOCs. Concerns have been raised concerning VOC loss from vacuum created in sample tubing.
	Bladder Pump	Bladder Pump	Least danger of VOC loss as entire sample train is under positive pressure and little sample heating occurs. Difficult to remove large volumes of water in reasonable time. Mild surging effect may keep turbidity elevated in sensitive wells.
Minimum-Purge, No-Purge Methods	------------------------------	------------------------------	Overall Method Comments- Advantages: Very little or no waste water. Well suited to repeat sampling events. Likely faster with lower costs. Disadvantages: Not directly equivalent to other methods. Vertical stratification or vertical flow conditions in the screened interval can result in deceptive or non-intuitive analytical results.
Predicated on the assumption that aquifer flow through the well maintains the water in the screened interval in a state equivalent to that in the aquifer. This assumption should be proven or the data qualified. Sampling is conducted with little or no purge, or by equilibrating a sampler in screened interval.	Pumps, various	Pumps, various	In the minimum-purge method, the internal volume of the sample tubing and pump is calculated. One volume of the pump and tubing is purged to flush the equipment and the well is then sampled.
	na	Passive Diffusion Bags	In most common form, a sealed water-filled polyethylene bag is allowed to equilibrate in the water column. Suitable primarily for VOCs. Generally require 2 week minimum in-situ residence time.
	na	Hydrasleeves	Collect a fixed volume of water from a specific interval. Requires duplicate samplers or redeployment for larger volumes. Sorbtion issues may bias results.
	na	Snap sampler	Deploys a sample container in the sampling interval where it is allowed to equilibrate (commonly for two weeks) before being sealed insitu by the sampler mechanism and retrieved. Limited to specific containers.

3.4 Multiple-Volume Purge

In the traditional Multiple-Volume Purge method, water is removed from the top of the water column, causing water to enter the screen and flush stagnant casing water upward to be subsequently removed. In recognition of the mixing of fresh and stagnant water in the casing section, a minimum of three well volumes is removed, at which time purging can be terminated upon parameter stabilization. Wells can be assumed to be adequately purged when five well volumes have been removed, although further purging may be conducted to meet specific goals, such as further reduction of turbidity.

3.4.1 Purge Volume Determination

Prior to initiating the purge, the amount of water standing in the water column (water inside the well riser and screen) should be determined The diameter of the well is determined and the water level and total depth of the well measured and recorded prior to inserting a pump or tubing into the well. The water level is subtracted from the total depth, providing the length of the water column. Specific methodology for obtaining these measurements is found in SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105).

Once this information is obtained, the volume of water to be purged can be determined using one of several methods. The well volume can be calculated using the equation:

$$V = 0.041\ d^2h$$

Where:
h = length of water column in feet
d = diameter of well in inches
V = one well volume in gallons

Alternatively, the volume of standing water in the well and the volume of three water columns may be determined using a casing volume per foot factor for the appropriate diameter well, such as *Table 2 Well Casing Diameter Volume Factors*. The water column length is multiplied by the appropriate factor in the Table 2 to determine the single well volume, three well volumes, or five well volumes for the well in question. Other acceptable methods include the use of nomographs or other equations or formulae.

TABLE 2, WELL CASING DIAMETER VOLUME FACTORS

	Reference	Minimum purge	Maximum purge*
	1 Well Volume (gallons/ft)	3 Well Volumes (gallons/ft)	5 Well Volumes (gallons/ft)
Well Casing Diameter (in)			
0.5	0.01	0.03	0.05
0.75	0.02	0.07	0.11
1	0.04	0.12	0.20
2	0.16	0.49	0.82
3	0.37	1.1	1.8
4	0.65	2.0	3.3
5	1.0	3.1	5.1
6	1.5	4.4	7.3
7	2.0	6.0	10.0
8	2.6	7.8	13.1
9	3.3	9.9	16.5
10	4.1	12.2	20.4
11	4.9	14.8	24.7
12	5.9	17.6	29.4
13	6.9	20.7	34.5
14	8.0	24.0	40.0
15	9.2	27.5	45.9
16	10.4	31.3	52.2
18	13.2	39.7	66.1
24	23.5	70.5	118
36	52.9	159	264
48	94.0	282	470

*** See text for discussion on terminating purge at five well volumes**

An adequate purge is normally achieved when three to five well volumes have been removed. The field notes should reflect the single well volume calculations or determinations, according to one of the above methods, and a reference to the appropriate multiplication of that volume, i.e., a minimum three well volumes, clearly identified as an initial purge volume goal.

3.4.2 Pumping Conditions

The pump or tubing inlet should be located at the top of the water column. If the pump is placed deep into the water column, the water above the pump may not be removed, and the subsequent samples, particularly if collected with a bailer, may not be representative of the aquifer conditions. If the recovery rate of the well is faster than the pump rate and no observable draw down occurs, the pump should be raised until the intake is as close as possible to the top of the water column for the duration of purging. If the pump rate exceeds the recovery rate of the well, the pump or tubing will have to be lowered to accommodate the drawdown.

3.4.3 Stability of Chemical Parameters

In the multiple-volume purge method, a stability determination may be made after three well volumes have been removed. If the chemical parameters have not stabilized according to the above criteria, additional well volumes (up to a total of five well volumes) should be removed. If the parameters have not stabilized after the removal of five well volumes, it is at the discretion of the project leader whether or not to collect a sample or to continue purging. If, after five well volumes, pH and conductivity have stabilized and the turbidity is still decreasing and approaching an acceptable level, additional purging should be considered to obtain the best sample possible.

3.4.4 Sample Collection

There are several means by which sampling can proceed after adequate volume has been purged and water quality parameters have stabilized. If a submersible pump and tubing are of suitable material and cleanliness for sample collection, sampling can proceed immediately by directly filling bottles from the tubing outlet. Commonly with the multiple-volume purge method, the pump is set up and cleaned in a manner suitable only for purging. In these cases, the pump is stopped and removed from the well and sampling proceeds with a bailer per the procedure described in Section 3.6.3. The pump should have a check valve to prevent water in the pump tubing from discharging back into the well when the pump is stopped. If a peristaltic pump is used, sampling can proceed as described in Section 3.6.1.

3.5 Low-Flow Method

This method involves placing the pump or tubing inlet within the screened interval of the well and purging at a low enough rate to achieve stable drawdown and minimal depression of the water level. The well is sampled without interruption after field parameters are stable and low turbidity is achieved. In general, only water in the screened interval of the well is pumped and the stagnant water in the well casing above the screen is not removed. Wells can generally be sampled in less time with less purge volume than with the multi-volume purge method. More attention is required in the assessment of stability criteria than the multi-volume method.

3.5.1 Nomenclature

A variety of terminology has been used to describe this method by SESD and others, including: ‘low flow’, ‘low-flow/low-volume’, ‘tubing-in-screen method’, ‘low flow/ minimal drawdown’, and ‘micropurge’. The current preferred SESD terminology for this method is ‘Low-Flow’. As the term ‘micropurge’ is sometimes used to refer to minimal-purge methods and has been trademarked by a vendor, the use of ‘micropurge’ to describe the Low-Flow method generally introduces ambiguity and confusion and thus the use of the term is discouraged.

3.5.2 Placement of Pump Tubing or Intake

The inlet of the pump tubing or intake of the submersible pump is placed in the approximate mid-portion of the screened interval of the well. While it is often thought that particular aquifer zones can be targeted by specific pump or intake placement, for conventionally constructed screened and filter-packed monitoring wells the zone monitored is only weakly dependent on the intake placement (Varljen, Barcelona, Obereiner & Kaminski, 2006).

The pump or tubing can be placed by carefully lowering them to the bottom of the well and then withdrawing half of the screen length, plus the length of any sump sections at the bottom of the well. A drawback of this approach is that it may stir up sediment at the well bottom. An alternate approach is to lower the pump or tubing a measured distance to place it at mid-screen without touching the bottom of the well. In the case of pumps, special care should be used in lowering them slowly, especially in the screened interval, to prevent elevating turbidity needlessly by the surging action of the pump.

3.5.3 Conditions of Pumping

Prior to initiation of pumping, a properly decontaminated well sounder should be lowered into the well to measure the water level prior to and during the purging process. Ideally, there should be only a slight and stable drawdown of the water column after pumping begins. In some cases, it will be necessary for the well to drawdown a considerable distance (10 ft or more in extreme cases) to maintain a minimal usable pumping rate for sampling (100-200 ml/min). Excessive pump rates and drawdown can result in increased turbidity, or aeration of the sample if the screen is exposed. Stable drawdown is an essential condition of the Low-Flow method. If the stable drawdown condition cannot be met, then one of the other methods should be employed.

3.5.4 Stability of Chemical Parameters

As with the Multiple-Volume Purging method described, it is important that all chemical parameters be stable prior to sampling. It is common for wells to require the removal of one of more screened-interval volumes (~2 gal for a 10 ft screen in a 2” dia. well) to achieve stability. Although it is possible for wells to achieve stability with lower purge volumes, the sampler should exercise caution in making an early stability determination.

3.5.5 Sample Collection

Low-Flow sampling is implemented using a pump and tubing suitable for sampling. After making the determination of parameter stability with stable drawdown, sampling can proceed immediately. Where submersible or bladder pumps are used, sampling can proceed by directly filling bottles from the tubing outlet. Where peristaltic pumps are used, sampling can proceed per the procedure described in Section 3.6.3.

3.6 Minimum-Purge and No-Purge Sampling

The Minimum-Purge and No-Purge sampling methods are employed when it is necessary to keep purge volumes to an absolute minimum, where it is desirable to reduce long-term monitoring costs, or where large wells or other limitations prevent well purging. The underlying assumption when employing these methods is that the water within the well screen is equilibrated with the groundwater in the associated formation. This assumption should be demonstrated prior to use of these methods or the results suitably qualified. These methods are generally impractical for SESD to implement because of the common lack of hydrogeological information in early investigative phases and the necessity with some methods that the samplers be pre-deployed to allow equilibration.

Vertical flow conditions and stratification of the water column have also been known to result in deceptive and non-intuitive analytical results. The use of these methods in the early phases of investigation can easily result in misinterpretation of site conditions and plume boundaries.

Particular caution is in order in the use of these methods when any of the following conditions exist:

- Low hydraulic conductivity ($K<10^{-5}$ cm/sec)
- Low groundwater surface gradients
- Fractured bedrock
- Wells with long screened intervals
- Wells screened in materials of varying hydraulic conductivities

If it is desired to transition a long-term monitoring program to Minimum-Purge or No-Purge sampling, a pilot study should be conducted where the Minimum-Purge or No-Purge sample results are compared to the conventional methods in use. Multiple samplers may be deployed in the screened interval to help establish appropriate monitoring intervals.

These methods are in common use and for the purposes of the SESD quality system they can be considered standard, but unaccredited, procedures. Several Minimum-Purge or No-Purge procedures that might be employed are shown below. It is not the intention to recommend particular equipment or vendors, and other equipment that can accomplish the same goals may be suitable.

3.6.1 Minimum Purge Sampling

The pump or tubing inlet is deployed in the screened interval. A volume of water equal to the internal pump and tubing volume is pumped to flush the equipment. Sampling then proceeds immediately. While superficially similar to Low-Flow sampling, the results obtained in this method will be sensitive to the vertical pump or tubing inlet placement and are subject to the limitations described above.

3.6.2 Passive Diffusion Bags

The no-purge Passive Diffusion Bag (PDB) typically consists of a sealed low-density polyethylene (LDPE) bag containing deionized water. They are deployed in the screened interval of a well and allowed to equilibrate, commonly for two weeks, prior to retrieval and decanting of the water into sample containers. Many volatile organic compounds will reach equilibrium across the LDPE material, including BTEX compounds and many chlorinated solvents. Compounds showing poor equilibration across LDPE include acetone, MTBE, MIBK, and styrene. PDBs have been constructed of other materials for sampling other analytes, but the vast majority of PDB samplers are of the LDPE material. Various vendors and the Interstate Technology and Regulatory Council (ITRC) can provide additional information on these devices.

3.6.3 HydraSleevesTM

HydraSleeeves™ are no-purge grab sampling devices consisting of a closed-bottom sleeve of low-density polyethylene with a reed valve at the top. They are deployed in a collapsed state to the desired interval and fill themselves through the reed valve when pulled upward through the sampling interval. The following is a summary of their operation:

> Sampler placement – A reusable weight is attached to the bottom of the sampler or the sampler is clipped to a weighted line. The HydraSleeve™ is lowered on the weighted line and placed with the top of the sampler at the bottom of the desired sampling interval. In-situ water pressure keeps the reed valve closed, preventing water from entering the sampler. The well is allowed to return to equilibrium.
>
> Sample collection - The reed valve opens to allow filling when the sampler is moved upward faster than 1 foot per second, either in one continuous upward pull or by cycling the sampler up and down to sample a shorter interval. There is no change in water level and only minimal agitation during collection.
>
> Sample retrieval - When the flexible sleeve is full, the reed valve closes and the sampler can be recovered without entry of extraneous overlying fluids. Samples are removed by puncturing the sleeve with the pointed discharge tube and draining the contents into containers for sampling or field parameter measurements.

Because the HydraSleeve™ is retrieved before equilibration can occur and they are constructed of non-Teflon® materials, there may be issues with sorbtion of contaminants in the use of this sampler.

3.6.4 Snap Samplers

The Snap Sampler is a patented no-purge groundwater sampling device that employs a double-end-opening bottle with "Snap" sealing end caps. The dedicated, device is deployed at the desired position in the screened interval with up to six Snap Samplers and six individual sampling bottles. The device is allowed to equilibrate in the screened interval and retrieved between 3 and 14 days after deployment. Longer deployments are possible to accommodate sampling schedules.

To operate, Snap Samplers are loaded with Snap Sampler bottles and the "Snap" caps are set into an open position. Samplers are deployed downhole with an attachment/trigger line and left to equilibrate downhole. To collect samples, the Snap Sampler bottles seal under the water surface by pulling a mechanical trigger line, or using an electric or pneumatic trigger system. The trigger releases Teflon® "Snap Caps" that seal the double-ended bottles. The end caps are designed to seal the water sample within the bottles with no headspace vapor. After the closed vial is retrieved from the well, the bottles are prepared with standard septa screw caps and labeled for laboratory submittal.

The manufacturer of the Snap Sampler provides considerable additional information on the validation and use of the device.

3.7 Equipment Considerations

Equipment choices are dictated by the purging and sampling method used, the depth to water, the quantity of water to be pumped, and quality considerations. The advantages and disadvantages of various commonly used pumps are discussed in the sections below and summarized in *Table 1, Purge and Sample Strategies with Equipment Considerations.* Additional information on the use of individual pumps is available in SESD Operating Procedure for Pump Operation, SESDPROC-203.

3.7.1 Use of Peristaltic Pumps

Peristaltic pumps are simple, inexpensive, and reliable equipment for purging and sampling where the limit of suction is not exceeded (approximately 25-30 vertical feet from the groundwater surface to the pump). When used for sampling, they should be equipped with new Teflon® tubing for each well. The flexible peristaltic pump-head tubing should also be changed between wells.

Samples for organic analyses cannot be exposed to the flexible peristaltic pump-head tubing, both due to the risk that the tubing would sorb contaminants and the propensity of this tubing to contribute organic compounds to the sample. Samples can be collected without contact with the pump-head tubing by the use of vacuum transfer caps for analyses requiring 1 liter glass containers and the use of the 'soda-straw' method for the filling of VOC vials.

The sample containers for the more turbidity-sensitive analyses are filled first, as filling the VOC vials (and to a lesser extent the glass bottles) may disturb the well and increase turbidity. The most appropriate order of sampling with a peristaltic pump is generally to fill poly containers for metals and classical analyses, followed by glass bottles for SVOCs and associated analyses, and finally to fill 40 ml VOC vials.

The following step-by-step procedure assumes that the pump has been set up per SESD Operating Procedure for Pump Operation (SESDPROC-203) and that containers for a typical full suite of analyses will be filled. The procedure is suitable for use with either multi-volume Purge and Low-Flow methods with minor differences in the collection of VOCs:

1. Deploy the lower end of the tubing to the desired point in the well. This would be the top-of-water for the multi-volume purge method or to the mid-screen for the Low-Flow method. Connect the well tubing to the flexible pump-head tubing and connect a short piece of tubing from the pump-head tubing to a measuring bucket.

2. Turn on the pump and establish a suitable pumping rate. For the multi-volume purge method, the rate will generally be a relatively fast rate that the well will sustain without elevating turbidity. For the low-flow method the pump rate is established at a slower rate to maintain a minimal and stable drawdown level.

3. Proceed with the measurement of water quality parameters and adjust the pump rate as needed to achieve low turbidity and stable drawdown.

4. When the well purge has been determined to be sufficient, fill containers for metals and classical analyses directly from the pump outlet. There is no need to interrupt pumping. The tubing should be held at the opening of the container and should not touch the container during filling. Protect caps from dust and debris during filling.

5. After filling the containers for metals and classical analyses stop the pump. Make sure that the tubing leading into the well is secured against movement during the following operations.

6. Create a crimp in the well tubing approximately one foot from the pump and grasp the crimped tubing in one hand. It is generally most effective to create a double 'Z' crimp.

7. Cut the sample tubing between the crimp and the pump. The tightly-held crimped tubing should keep water from running back into the well. In lieu of

cutting the tubing, the well tubing can be disconnected from the pump and a short piece of tubing connected in its place.

8. Insert both free ends of the tubing into the ferrule-nut fittings of a pre-cleaned Teflon® transfer cap assembly and tighten the nuts. Attach the transfer cap assembly to the first glass container for semi-volatile analysis and securely tighten the threaded ring.

9. Turn the pump on. Very slowly release the 'Z' crimp in the sample tubing. As vacuum builds up in the sample container, water should begin to move up the sample tubing instead of back into the well. If after several minutes water has not begun moving up the tubing, check the tightness of fittings and the attachment of the cap to the bottle. Allowing water to rush back down the tubing from the 'Z' crimp can surge the well and elevate turbidity.

10. Fill the container to about halfway between the shoulder and the neck. Crimp the well tubing. Move the transfer cap to any additional bottles and repeat the filling process.

11. When finished filling bottles with the transfer cap, again crimp the tubing. Remove the well tubing from the transfer cap and reattach it to the pump. Slowly run the pump and release the crimp until water is approaching the flexible peristaltic tubing.

12. Make a kink or otherwise mark the tubing at the top of the casing in case the tubing needs to be reinserted for additional sample volume. Slowly remove the tubing from the well and coil it in one hand in loose coils. With the top end of the tubing blocked, water is retained in the tubing as it is withdrawn, much as in a capped soda straw, hence the name for this method.

13. Remove the top from a 40 ml VOC vial and position the end of the sample tubing near the top of the vial. Reverse the pump direction and turn the speed knob to its slowest position. Turn on the pump and slowly increase speed until water slowly fills the vial. Fill the vial with a slow laminar flow that does not agitate the water in the vial or entrain bubbles. Continue to fill the vial until a convex meniscus forms on the top of the vial and turn off the pump.

14. Carefully screw the septum-lid to the vial and fasten firmly. Invert the vial and tap on your knuckles to check for bubbles. Carefully add additional volume to the vial if necessary. Small bubbles are undesirable but may be unavoidable with some media, especially when using pre-preserved vials.

15. Repeat the filling process for additional vials. Avoid partially filling vials as the available water in the tubing is used. If more volume is required than that contained in the tubing, purge the remaining water from the tubing and reinsert

the tubing in the well to the level marked previously. Run the pump to refill the tubing. If performing Low-Flow sampling, run additional volume through the pump to purge any water that may have been collected from the stagnant water column.

16. Fill additional vials as needed. Be sure that any water that has contacted the flexible peristaltic tubing is not pumped into a vial.

3.7.2 Use of Submersible Centrifugal Pumps

Submersible centrifugal pumps are used in wells of 2" diameter and larger. They are especially useful where large volumes of water are to be removed or when the groundwater surface is a large distance below ground surface. Commonly used pumps are the Grundfos® Redi-Flo2, the Geotech GeoSub, and the various 'Monsoon' style pumps. Other pumps are acceptable if constructed of suitable materials.

When used with the Multiple-Volume Purge method, the pump is generally used only to purge, with sampling performed with a bailer. In this use, the pump can be used with polyethylene or other tubing or hose that will not contribute contaminants to the well. The pump and tubing is decontaminated between wells per the relevant provisions of SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205). When used in this application the pump should be equipped with a check valve to prevent water in the discharge tubing or hose from running back down into the well.

When used for Low-Flow purging and sampling the pump must be constructed of stainless steel and Teflon®. Pump cleaning at each well follows the more stringent procedures described in SESD Operating Procedure for Field Equipment Cleaning and Decontamination SESDPROC-205) for this application. The sample tubing should be either new Teflon® tubing, or tubing dedicated to each well. Dedicated tubing would ideally be cleaned between uses, but tubing stored in the well casing between uses is acceptable, although caution should be exercised where very high concentrations of contaminants have been sampled in a well.

3.7.3 Use of Bailers

Bailers are a common means of sampling when the Multiple-Volume Purge method is used. They are occasionally used for purging when other equipment is not available or has failed. As bailers surge the well on each withdrawal, it is very difficult to lower turbidity adequately during a well purge, and when used for sampling they can elevate turbidity in a well before all sample volume is collected. If not lowered carefully into the top of the water column, the agitation may strip volatile compounds. Due to the difficulties and limitations inherent in their use, other sampling or purging means should generally be given preference.

Bailers should be closed-top Teflon® bailers with Teflon® coated stainless steel leaders used with new nylon haul rope. They are lowered gently into the top of the water column, allowed to fill, and removed slowly. It is critical that bailers be slowly and gently immersed into the top of the water column, particularly during final stages of purging and during sampling, to minimize turbidity and loss of volatile organic constituents.

If the well has previously been purged with a pump, there is likely stagnant water at the top of the well that was above the pump or tubing inlet. Several bailers of water should be retrieved and discarded to assure the upper stagnant water has been removed.

When sampling, containers are filled directly by pouring from the outlet at the top of the bailer. Containers for metals analysis should be filled first in case the bailing process increases well turbidity. VOC vials should be filled carefully and slowly with a laminar flow to reduce agitation and the stripping of VOCs.

3.7.4 Use of Bladder Pumps

Bladder pumps use a source of compressed gas to compress and release a bladder straddled by check valves within the pump body. As the bladder is compressed, water is expelled out the upper check valve to the surface. When gas pressure is released, the bladder refills as well water enters the lower pump inlet. A control unit is used to control the pressure and timing of the bladder inflation gas flow.

Bladder pumps are capable of pumping from moderate depths to water, but are not capable of high flow rates. As they operate cyclically, the well is surged slightly on each cycle and it may be difficult to lower turbidity in sensitive or poorly developed wells. As the entire sample train is under positive pressure and the pumps develop little heat, they are ideal for sampling VOCs.

Prior to sampling and between each well the pumps are cleaned internally and externally per the provisions of SESD Operating Procedure for Field Decontamination (SESDPROC-205) and a new Teflon® bladder installed. New (or dedicated) Teflon® sample tubing is used at each well, although polyethylene tubing can be used for the compressed gas drive line and cleaned between each well.

3.7.5 Use of Inertial Pumps

Inertial pumps consist of a check valve which is affixed to the lower end of semi-rigid tubing. The tubing and valve are cycled up and down, allowing water to alternately be drawn into the check valve inlet and then pulled up towards the surface. Two commonly used inertial pumps are the Waterrra® pump for wells 1arger than 1” and the Geoprobe® Tubing Check Valve for small diameter wells. The primary use of these pumps is in well development where their near-immunity to silt is an advantage. Inertial pumps should not be used for the final well purge or for sampling as there is a low likelihood of reducing turbidity to appropriate levels and they have the potential to strip volatiles from the water column through agitation.

To set up the pump, the check valve is screwed onto the discharge tubing where it will cut its own threads. In the case of the Waterra® pump, a surge block can also be pressed onto the check valve. The pump is lowered into the well to the screened interval and rapidly cycled up and down a distance of 3” -12”. The stroke length and speed are adjusted for pumping effect. Electric actuators can be used to reduce the effort involved. The pump should be moved to different levels in the screen to surge the entire screen. The pump can occasionally be lowered to the bottom of the well to vacuum out silt. Any silt that clogs the valve is usually quickly rinsed out by the pump cycling and if the clog remains the pump is easily retrieved and redeployed.

The surging activity is usually continued until turbidity is lowered to a measurable range and cannot easily be lowered further. Further development or purging is then conducted with other pumps.

3.8 Wells With In-Place Plumbing

Wells with in-place plumbing are commonly found at municipal water treatment plants, industrial water supplies, private residences, and in other applications. Many permanent monitoring wells at active facilities are also equipped with dedicated, in-place pumps.

A permanent monitoring well with an in-place pump may be treated as other monitoring wells without pumps. Since the in-place pump is generally “hard” mounted at a pre-selected depth, it cannot be moved up or down during purging and sampling. If the pump inlet is above the screened interval, the well should be sampled using the Multiple-Volume Purge method. If the pump intake is located within the screened interval, the well can be sampled using Low-Flow procedures. Known details of pump type and construction, tubing types, pump setting depths, and any other available information about the system should be recorded in the field logbook.

In the case of the other types of wells, e.g., municipal, industrial and residential supply wells, there is typically not enough known about the construction aspects of the wells to apply the same criteria as used for monitoring wells. The volume to be purged in these situations therefore depends on several factors: whether the pumps are running continuously or intermittently and whether or not any storage/pressure tanks are located between the sampling point and the pump. The following considerations and procedures should be followed when purging wells with in-place plumbing under the conditions described.

3.8.1 Continuously Running Pumps

If the pump runs more or less continuously, no purge (other than opening a valve and allowing it to flush for a few minutes) is necessary. If a storage tank is present, a spigot, valve or other sampling point should be found located between the pump and the storage tank. If no valve is present, locate and use the valve closest to the tank. Measurements of field parameters are recorded immediately prior to the time of sampling.

3.8.2 Intermittently or Infrequently Running Pumps

If the pump runs intermittently or infrequently, best judgment should be utilized to remove enough water from the plumbing to flush standing water from the piping and any storage tanks that might be present. Often under these conditions, 15 to 30 minutes of purging will be adequate. Measurements of pH, specific conductance, temperature and turbidity should be made and recorded at intervals during the purge and the final measurements made at the time of sampling should be considered the measurements of record for the event.

3.9 Temporary Monitoring Wells

3.9.1 General Considerations

As temporary wells are installed for immediate sample acquisition, the procedures used to purge temporary ground water monitoring wells may differ from those for permanent wells. Temporary wells include standard well screen and riser placed in boreholes created by hand augering or drilling, or they may consist of a drive rod and screen such as a direct-push Geoprobe® Screen Point that is driven into place at the desired sampling interval. As aquifer water enters the sampler immediately upon deployment, the requirement to remove several volumes of water to replace stagnant water does not necessarily apply. In practice, developing and purging the well to usable turbidity levels will remove many times the water that would be removed in a Multiple-Volume Purge with calculated well volumes. It is important to note, however, that the longer a temporary well is in place and not sampled, the more stagnant the water column becomes and the more appropriate it becomes to apply standard permanent monitoring well purging criteria to achieve representative aquifer conditions in the sample.

3.9.2 Development of Temporary Wells

In cases where the temporary well is to be sampled immediately after installation, purging is conducted primarily to mitigate the impacts of installation. In most cases, temporary well installation procedures disturb the existing aquifer conditions, causing extreme turbidity. The goal of purging is to reduce the turbidity and remove the volume of water in the area directly impacted by the installation procedure.

The following procedure has been found to be effective in developing and sampling small diameter temporary wells where a peristaltic pump can be used. Turbidity can generally be lowered to 50 NTU at the time of sampling and turbidity less than 10 NTU is often achieved.

1. Cut peristaltic tubing to reach to the bottom of the well. Connect to a peristaltic pump and begin pumping at a high rate.

2. Use the tubing to vacuum out sediment at the bottom of the well.

3. Aggressively surge the end of the tubing in the screened interval by cycling the tubing rapidly up and down. Periodically repeat vacuuming of the well bottom.

4. When a visible 'break' to a lower turbidity is observed, cease surging the well and begin lowering the pumping rate.

5. When the water clears (turbidity < 100-200 NTU) begin raising the end of the tubing to the top of the water column.

6. Continue purging from the top of the water column, lowering the pump speed as required to lower turbidity. When adequately low turbidity and stable water quality parameters have been achieved, sampling can proceed.

Where the water level is below the limit of suction in a small diameter temporary well, a Geoprobe® mechanical bladder pump can be used for purging and sampling. The well should first be developed with an inertial pump to remove the bulk of silt and suspended particles that could clog the check valves of the bladder pump. The inertial pump is used to vacuum out the bottom of the well and surged in the screened interval until a 'break' to lower turbidity is observed prior to deployment of the bladder pump. Since the mechanical bladder pump requires cumbersome redeployment to change its pumping level, it should be deployed low enough in the water column that the water level will not be lowered below the pump during purging and sampling. The mechanical bladder pump is generally deployed above the screened interval to facilitate the settling of particles, but below the top of the water column to alleviate the need to reset the pump. Detailed instructions on the deployment of the pump can be found in SESDPROC203, Pump Operation.

3.9.3 Decommissioning of Temporary Wells

After temporary wells have fulfilled their purpose, they should be properly decommissioned similar to permanent wells. In general, the casings and screens can be easily removed and the borehole should then be pressure grouted from the bottom of the original borehole to prevent surface contamination of the aquifer, cross-connection of aquifers, and to remove a potential vapor pathway.

Direct-push screen-point wells may be decommissioned by one of two methods.

1. A disposable screen is used. The sampling sheath is pulled off of the screen and a 30% solids bentonite grout is pumped down the tool string as the rods are withdrawn.

Grout volumes are measured during pumping to assure that the hole is completely filled. The disposable screen is left behind at the bottom of the borehole.

2. The screen is removed with the sampler sheath and tool string. The hole is immediately re-entered with an empty sample sheath with disposable point. Upon reaching the original total depth of the temporary well, 30% solids bentonite grout is pumped down the tool string with the pumped volume monitored during tool string withdrawal to assure that the hole is completely filled.

A system is available to insert a small diameter grouting tube down through the screen-point screen. Grout is pumped through the grouting tube while the tools are withdrawn. SESD does not use this system as grout denser than 20% solids cannot reliably be installed with this system.

Additional guidance on decommissioning may be found in SESDGUID-101, Design and Installation of Monitoring Wells.

3.9.4 Other Considerations for Direct-Push Groundwater Sampling

With certain direct push sampling techniques, such as the Hydropunch™ and other discrete samplers used with cone-penetrometer rigs, purging is either not practical or not possible. The sampling device is simply pushed or driven to the desired depth and opened, whereupon the sample is collected and retrieved. As a result, some samples collected in this way may not be satisfactory or acceptable for certain analyses, i.e., the sampler may collect a turbid sample inappropriate for metals analyses or the sample may have inadequate volume to achieve desired reporting levels.

3.10 Wells Purged to Dryness

In some situations, even with slow purge rates, a well may be purged dry in the Multiple-Volume Purge method or stable drawdown cannot be maintained in the Low-Flow method. In these cases, the well should be purged to dryness (evacuated) and sampled upon recovery of adequate volume for sampling. Sampling should occur as soon as adequate volume has recovered. The field parameters should be measured and recorded at the time of sample collection as the measurements of record for the sampling event.

Sampling under these conditions is not ideal and suitable qualifications of the data should be included in the report. Water cascading down the screen into the well may strip volatile compounds and elevate turbidity. Although suffering from other limitations, No-Purge methods may prove useful for these wells.

4 Additional Purging and Sampling Considerations

4.1 Field Care of Purging Equipment

New plastic sheeting should be placed on the ground surface around the well casing to prevent contamination of the pumps, hoses, ropes, etc., in the event they accidentally come into contact with the ground surface or, for some reason, they need to be placed on the ground during the purging event. It is preferable that hoses used in purging that come into contact with the ground water be kept on a spool or contained in a large wash tub lined with plastic sheeting, both during transportation and during field use, to further minimize contamination by the transporting vehicle or the ground surface.

Careful consideration shall be given to using submersible centrifugal or bladder pumps to purge wells which are excessively contaminated with oily compounds as it may be difficult to adequately decontaminate severely contaminated pumps under field conditions. When wells of this type are encountered, alternative equipment, such as bailers or peristaltic pumps, should be considered.

4.2 Investigation Derived Waste

Purging and field cleaning of equipment generates liquid investigation derived waste (IDW), the disposition of which must be considered. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for guidance on management or disposal of this waste.

4.3 Sample Preservation

After sample collection, all samples requiring preservation must be preserved as soon as practical. Consult the Analytical Services Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM) for the correct preservative for the particular analytes of interest. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

4.4 Special Sample Collection Procedures

4.4.1 Trace Organic Compounds and Metals

Special sample handling procedures should be instituted when trace contaminant samples are being collected. All sampling equipment, including pumps, bailers, water level measurement equipment, etc., which contacts the water in the well must be cleaned in accordance with the cleaning procedures described in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-

206). Pumps should not be used for sampling unless the interior and exterior portions of the pump and the discharge hoses are thoroughly cleaned. Rinse blank samples should be collected to verify the adequacy of cleaning when using a sampling pump other than a peristaltic pump.

4.4.2 Order of Sampling with Respect to Analytes

In many situations when sampling permanent or temporary monitoring wells, sufficiently low turbidity is difficult to achieve and maintain. Removal and insertion of equipment after the purge or during sampling may negate the low turbidities achieved during purging and elevate turbidity back to unacceptable levels. For this reason, it is important that special efforts be used to minimize any disturbance of the water column after purging and to fill sample containers for metals analysis first. The preferred order of sampling is metals first, followed by other inorganic analytes, extractable organic compounds, and finally volatile organic compounds.

4.5 Filtering

As many contaminants are known to sorb to soil particles, the normal goal of sampling is to reduce the presence of these particles (measured by turbidity) in order that the dissolved concentration of contaminants can be obtained. However, transport of sorbed contamination on colloidal particles can be a means of contaminant transport on some sites. For this reason, the SESD approach is to reduce turbidity through the careful purging of wells, rather than through filtering of samples, in order that the colloidal particles would be included in the sample.

As a standard practice, ground water samples will not be filtered for routine analysis. Filtering will usually only be performed to determine the fraction of major ions and trace metals passing the filter and used for flow system analysis and for the purpose of geochemical speciation modeling. Filtration is not acceptable to correct for improperly designed or constructed monitoring wells, inadequate well development, inappropriate sampling methods, or poor sampling technique.

When samples are collected for routine analyses and are filtered, both filtered and non-filtered samples will be submitted for analyses. Samples for organic compounds analysis should not be filtered. Prior to filtration of the ground water sample for any reason other than geochemical speciation modeling, the following criteria must be demonstrated to justify the use of filtered samples for inorganic analysis:

1. The monitoring wells, whether temporary or permanent, have been constructed and developed in accordance with the SESD Guidance Document, Design and Installation of Monitoring Wells (SESDGUID-001).

2. The ground water samples were collected using sampling techniques in accordance with this section, and the ground water samples were analyzed in accordance with USEPA approved methods.

3. Efforts have been undertaken to minimize any persistent sample turbidity problems. These efforts may consist of the redevelopment or re-installation of permanent ground water monitoring wells or the implementation of carefully conducted low flow rate sampling techniques.

If filtration is necessary for purposes of geochemical modeling or other **pre-approved** cases, the following procedures are suggested:

1. Accomplish in-line filtration through the use of disposable, high capacity filter cartridges (barrel-type) or membrane filters in an in-line filter apparatus. The high capacity, barrel-type filter is preferred due to the higher surface area associated with this configuration. If a membrane filter is utilized, a minimum diameter of 142 mm is suggested.

2. When using pumps for sampling, the filter can generally be attached directly to the pump outlet. When sampling with a bailer or when otherwise required, an initial unfiltered sample with extra volume will be collected, and a peristaltic pump with filter used to decant and filter the sample to the final sample container.

3. Use a 0.45 µm pore-size filter to remove most non-dissolved particles. A 5 µm or 10 µm pore-size filter should be used for the purpose of determining colloidal constituent concentrations.

4. Fill the filter and rinse with approximately one additional filter volume prior to filling sample bottles

Potential differences can result from variations in filtration procedures used to process water samples for the determination of trace element concentrations. A number of factors associated with filtration can substantially alter "dissolved" trace element concentrations; these include filter pore size, filter type, filter diameter, filtration method, volume of sample processed, suspended sediment concentration, suspended sediment grain-size distribution, concentration of colloids and colloidally-associated trace elements, and concentration of organic matter. Therefore, consistency is critical in the comparison of short-term and long-term results. Further guidance on filtration may be obtained from the following: 1) Metals in Ground Water: Sampling Artifacts and Reproducibility; 2) Filtration of Ground Water Samples for Metals Analysis; and 3) Ground Water Sampling - A Workshop Summary. See Section 1.4, References, for complete citation for these documents.

4.6 Bacterial Sampling

Whenever wells (normally potable wells) are sampled for bacteriological parameters, care must be taken to ensure the sterility of all sampling equipment and all other equipment entering the well. Further information regarding bacteriological sampling is available in the following: 1) Sampling for Organic Chemicals and Microorganisms in the Subsurface; 2) Handbook for Evaluating Water Bacteriological Laboratories; and 3) Microbiological Methods for Monitoring the Environment, Water and Wastes. See Section 1.4, References, for complete citation for these documents.

4.7 Specific Sampling Equipment Quality Assurance Techniques

All equipment used to collect ground water samples shall be cleaned as outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206). Malfunctioning equipment should be labeled in the field and repaired, before being stored at the conclusion of field studies. Cleaning procedures utilized in the field or field repairs shall be thoroughly documented in field records.

4.8 Auxiliary Data Collection

During ground water sample collection, it is important to record a variety of ground water related data. Included in the category of auxiliary data are water levels measured according to the SESD Operating Procedure for Groundwater Level and Well Depth Measurement (SESDPROC-105), well volume determinations, pumping rates during purging, and, driller or boring logs. This information should be documented in the field records.

4.9 Well Development

Wells may be encountered that are difficult to sample effectively due to inadequate initial development or the need for redevelopment due to scaling, sedimentation, corrosion, or biofouling. These wells may produce water only at low flow rates or water with chronically elevated turbidity. Redevelopment of these wells should be considered as the process can improve sample quality and speed field operations. Well development procedures are described in Design and Installation of Monitoring Wells (SESDGUID-101).

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Sediment Sampling**	
Effective Date: August 21, 2014	**Number:** SESDPROC-200-R3
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-200-R3, *Sediment Sampling*, replaces SESDPROC-200-R2. **General:** Corrected any typographical, grammatical, and/or editorial errors. Throughout the document mention of quality system or SESD quality system was replaced with Field Branches Quality System or FBQS. **Cover Page:** Changed the Enforcement and Investigations Branch Chief from Archie Lee to Acting Chief, John Deatrick. Changed the Ecological Assessment Branch Chief from Bill Cosgrove to Acting Chief, Laura Ackerman. Changed the FQM from Liza Montalvo to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. Throughout the document: any reference to “Percent Moisture” was changed to “Percent Solids.”	August 21, 2014
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SESDPROC-200-R0, *Sediment Sampling*, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting sediment samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling sediment samples in the field. On the occasion that SESD field investigators determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to obtain a sediment sample, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Control of Records, SESDPROC-004, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-104, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

United States Office of Occupational Health and Safety (US OSHA). 1981. Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples (PM-273), Memo from David Weitzman, Work Group Chairman, US EPA. April 13, 1981.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting sediment samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting sediment samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and stored in a secure place.

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2 Special Sampling Considerations

2.1 Sediment Samples for Volatile Organic Compounds Analysis

If samples are to be analyzed for volatile organic compounds (VOCs), they should be collected in a manner that minimizes disturbance of the sample. The sample for VOC analysis should be collected directly from the sample device, if possible, before it is emptied into the pan. It may not be possible to do this with certain types of sediment sampling equipment, such as the Ponar dredge. In cases such as these, the VOC aliquots should be collected from the dredge contents immediately after they have been deposited in the pan and prior to any mixing. The sample shall be placed in the appropriate container (En Core® Sampler or other Method 5035 compatible container) with no headspace. ***Samples for VOC analysis are not homogenized***. Preservatives may be required for some samples with certain variations of Method 5035. Consult the method description below in Section 2.2, Sediment Sampling (Method 5035) or the principal analytical chemist to determine if preservatives are necessary.

In some cases, the sediment may be soft and not lend itself to collection by plunging En Core® Samplers or syringe samplers into the sample matrix. In these cases, it is appropriate to open the sample device, i.e., the En Core® Sampler barrel or syringe, prior to sample collection, and to carefully place the sediment in the device, filling it fully with the required volume of sample.

2.2 Sediment Sampling (Method 5035)

The following sampling protocol is recommended for site investigators assessing the extent of VOCs in sediments at a project site. Because of the large number of options available, careful coordination between field and laboratory personnel is needed. The specific sampling containers and sampling tools required will depend upon the detection levels and intended data use. Once this information has been established, selection of the appropriate sampling procedure and preservation method best applicable to the investigation can be made.

2.2.1 Equipment

Sediment for VOC analyses may be retrieved using any of the SESD sediment sampling methods described in Sections 3 through 6 of this procedure. Once the sediment has been obtained, the En Core® Sampler, syringes, stainless steel spatula, standard 2-oz. sediment VOC container, or pre-prepared 40 ml vials may be used/required for sub-sampling. The specific sample containers and the sampling tools required will depend upon the data quality objectives established for the site or sampling investigation. The various sub-sampling methods are described below.

2.2.2 Sampling Methodology - Low Concentrations

When the total VOC concentration in the sediment is expected to be less than 200 µg/kg, the samples may be collected directly with the En Core® Sampler or syringe. If using the syringes, the sample must be placed in the sample container (40 ml pre-prepared vial) immediately to reduce volatilization losses. The 40 ml vials should contain 10 ml of organic-free water for an un-preserved sample or approximately 10 ml of organic-free water and a preservative. It is recommended that the 40 ml vials be prepared and weighed by the laboratory (commercial sources are available which supply preserved and tared vials). When sampling directly with the En Core® Sampler, the vial must be immediately capped and locked.

A sediment sample for VOC analysis may also be collected with conventional sampling equipment. A sample collected in this fashion must either be placed in the final sample container (En Core® Sampler or 40 ml pre-prepared vial) immediately or the sample may be immediately placed into an intermediate sample container with no head space. If an intermediate container (usually 2-oz. sediment jar) is used, the sample must be transferred to the final sample container (En Core® Sampler or 40 ml pre-prepared vial) as soon as possible, not to exceed 30 minutes.

NOTE: After collection of the sample into either the En Core® Sampler or other container, the sample must immediately be stored in an ice chest and cooled.

Sediment samples may be prepared for shipping and analysis as follows:

En Core® Sampler - the sample shall be capped, locked, and secured in a plastic bag.

Syringe - Add about 3.7 cc (approximately 5 grams) of sample material to 40-ml pre-prepared containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag. Note: When using the syringes, it is important that no air is allowed to become trapped behind the sample prior to extrusion, as this will adversely affect the sample.

Stainless Steel Laboratory Spatulas - Add between 4.5 and 5.5 grams (approximate) of sample material to 40 ml containers. Secure the containers in a plastic bag. Do not use a custody seal on the container; place the custody seal on the plastic bag.

2.2.3 Sampling Methodology - High Concentrations

Based upon the data quality objectives and the detection level requirements, this high level method may also be used. Specifically, the sample may be packed into a single 2-oz. glass container with a screw cap and septum seal. The sample container must be filled quickly and completely to eliminate head space. Sediments containing high total VOC concentrations may also be collected as described in Section 2.2.2, Sampling Methodology - Low Concentrations, and preserved using 10 ml methanol.

2.2.4 Special Techniques and Considerations for Method 5035

Effervescence

If low concentration samples effervesce from contact with the acid preservative, then either a test for effervescence must be performed prior to sampling, or the investigators must be prepared to collect each sample both preserved or un-preserved as needed, or all samples must be collected unpreserved.

To check for effervescence, collect a test sample and add to a pre-preserved vial. If preservation (acidification) of the sample results in effervescence (rapid formation of bubbles) then preservation by acidification is not acceptable, and the sample must be collected un-preserved.

If effervescence occurs and only pre-preserved sample vials are available, the preservative solution may be placed into an appropriate hazardous waste container and the vials triple rinsed with organic-free water. An appropriate amount of organic-free water, equal to the amount of preservative solution, should be placed into the vial. The sample may then be collected as an un-preserved sample. Note that the amount of organic free water placed into the vials will have to be accurately measured.

Sample Size

While this method is an improvement over earlier ones, field investigators must be aware of an inherent limitation. Because of the extremely small sample size, sample representativeness for VOCs may be reduced compared to samples with larger volumes collected for other constituents. The sampling design and objectives of the investigation should take this into consideration.

Holding Times

Sample holding times are specified in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version. Field investigators should note that the holding time for an un-preserved VOC sediment sample is 48 hours. Arrangements should be made to ship the sediment VOC samples to the laboratory by overnight delivery the day they are collected so the laboratory may preserve and/or analyze the sample within 48 hours of collection.

Percent Solids

Samplers must ensure that the laboratory has sufficient material to determine percent solids in the VOC sediment sample to correct the analytical results to dry weight. If other analyses requiring percent solids determination are being performed upon the sample, these results may be used. If not, a separate sample (minimum of 2 oz.) for percent solids determination will be required.

Safety

Methanol is a toxic and flammable liquid. Therefore, methanol must be handled with all required safety precautions related to toxic and flammable liquids. Inhalation of methanol vapors must be avoided. Vials should be opened and closed quickly during the sample preservation procedure. Methanol must be handled in a ventilated area. Use protective gloves when handling the methanol vials. Store methanol away from sources of ignition such as extreme heat or open flames. The vials of methanol should be stored in a cooler with ice at all times.

Shipping

Methanol and sodium bisulfate are considered dangerous goods, therefore shipment of samples preserved with these materials by common carrier is regulated by the U.S. Department of Transportation and the International Air Transport Association (IATA). The rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179) and the current edition of the IATA Dangerous Goods Regulations must be followed when shipping methanol and sodium bisulfate. Consult the above documents or the carrier for additional information. Shipment of the quantities of methanol and sodium bisulfate used for sample preservation falls under the exemption for small quantities. A summary of the requirements for shipping samples follows. Refer to the code for a complete review of the requirements.

1. The maximum volume of methanol or sodium bisulfate in a sample container is

limited to thirty (30) ml.

2. The sample container must not be full of methanol.

3. The sample container must be stored upright and have the lid held securely in place. Note that the mechanism used to hold the cap in place must be able to be completely removed so weight is not added to the sample container, as specified in Method 5035.

4. Sample containers must be packed in an absorbent material capable of absorbing spills from leaks or breakage of the sample containers.

5. The maximum sample shuttle weight must not exceed 64 pounds.

6 The maximum volume of methanol or sodium bisulfate per shipping container is 500 ml.

7 The shipper must mark the sample shuttle in accordance with shipping dangerous goods in acceptable quantities.

8. The package must not be opened or altered until no longer in commerce.

The following summary table lists the options available for compliance with SW846 Method 5035. The advantages and disadvantages are noted for each option. SESD's goal is to minimize the use of hazardous material (methanol and sodium bisulfate) and minimize the generation of hazardous waste during sample collection.

Table 1: Method 5035 Summary

OPTION	PROCEDURE	ADVANTAGES	DISADVANTAGES
1	Collect 2 – 40 ml vials with ~5 grams of sample and 1 – 2 oz. glass w/septum lid for screening and % solids	Screening conducted by lab	Presently a 48 hour holding time for unpreserved samples
2	Collect 3 EnCore® Samplers and 1 – 2oz. glass w/septum lid for screening and % solids	Lab conducts all preservation/preparation procedures	Presently a 48 hour holding time for preparation of samples
3	Collect 2 – 40 ml vials with 5 grams of sample and preserve w/methanol or sodium bisulfate, and 1 – 2 oz. glass w/septum lid for screening and % solids	High level VOC samples may be composited Longer holding time	Hazardous materials used in field
4	Collect 1 – 2 oz. glass w/septum lid for analysis and % solids	Lab conducts all preservation/preparation procedures	May have significant VOC loss

2.3 Special Precautions for Trace Contaminant Sediment Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers with samples suspected of containing high concentrations of contaminants shall be stored separately. All background samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected contaminated area if sampling devices are to be reused. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.
- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.

- Samplers must use new, verified and certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, or SESD Operating Procedure for Field Cleaning and Decontamination at the FEC, SESDPROC-206, for collection of samples for trace metals or organic compound analyses.

2.4 Sample Homogenization

1. If sub-sampling of the primary sample is to be performed in the laboratory, transfer the entire primary sample directly into an appropriate, labeled sample container(s). Proceed to step 5

2. If sub-sampling the primary sample in the field or compositing multiple primary samples in the field, place the sample into a glass or stainless steel homogenization container and mix thoroughly. Each aliquot of a composite sample should be of the same volume.

3. All sediment samples must be thoroughly mixed to ensure that the sample is as representative as possible of the sample media. ***Samples for VOC analysis are not homogenized.*** The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

    - The material in the sample pan should be divided into quarters and each quarter should be mixed individually.
    - Two quarters should then be mixed to form halves.
    - The two halves should be mixed to form a homogenous matrix.

    This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction, and occasionally turning the material over.

4. Place the sample into an appropriate, labeled container(s) using the alternate shoveling method and secure the cap(s) tightly. Threads on the container and lid should be cleaned to ensure a tight seal when closed.

5. Return any unused sample material back to the location from which the sample was collected.

2.5 Quality Control

If possible, a control sample should be collected from an area not affected by the possible contaminants of concern and submitted with the other samples. The control sample should be collected at an upstream location in the same stream or conveyance from which the primary samples area collected. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by sampling tools.

2.6 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-004. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Procedure for Sample and Evidence Management, SESDPROC-005.

3 General Considerations

3.1 General

The sediment sampling techniques and equipment described in the following Sections 4, 5 and 6 of this procedure document are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in this section are followed, a representative sample of the sediment should be obtained.

3.2 Equipment Selection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. Wading is the preferred method for reaching the sampling location, particularly if the stream has a noticeable current (is not impounded). However, wading may disrupt bottom sediments causing biased results; therefore, the samples should be collected facing upstream. If the stream is too deep to wade, the sediment sample may be collected from a platform such as a boat or a bridge.

To collect a sediment sample from a water body or other surface water conveyance, a variety of methods can be used:

- Scoops and spoons
- Dredges (Ponar, Young)
- Coring Devices (tubes, Shelby tubes, Ogeechee Sand Pounders®, and augers)
- Vibracore® (Electronic Vibratory Core Tube Driver)

Regardless of the method used, precautions should be taken to insure that the sample collected is representative of the water body or conveyance. These methods are discussed in the following paragraphs.

4 Stainless Steel Scoops and Spoons

4.1 Wading

If the conveyance is dry or is a wadeable surface water body, the easiest way to collect a sediment sample is by using a stainless steel scoop or spoon. If the conveyance is dry, the sediment is accessed directly and is collected using either the stainless steel scoop or spoon. If the conveyance is a wadeable stream or other water body, the method is accomplished by wading into the surface water body and while facing upstream (into the current), scooping the sample along the bottom of the surface water body in the upstream direction. Excess water may be removed/drained from the scoop or spoon. However, this may result in the loss of some fine-grained particle size material associated with the substrate being sampled. Care should be taken to minimize the loss of this fine-grained material. Aliquots of the sample thus collected are then placed in a glass pan and homogenized according to the quartering method described in Section 2.4.

4.2 Bank/Platform Sampling

In surface water bodies that are too deep to wade, but less than eight feet deep, a stainless steel scoop or spoon attached to a piece of conduit can be used either from the banks, if the surface water body is narrow, or from a boat. Again, care should be taken to minimize the loss of the fine particle sizes. The sediment is placed into a glass pan and mixed according to the quartering method described in Section 2.4.

5 Dredges

5.1 General Considerations

Dredges provide a means of collecting sediment from surface water bodies that are too deep to access with a scoop and conduit. They are most useful when collecting softer, finer-grained substrates comprised of silts and clays but can also be used to collect sediments comprised of sands and gravel, although sample recovery in these materials may be less than complete.

Free, vertical clearance is required to use any of the dredges. Dredges, attached to ropes, are lowered vertically from the sampling platform (boat, bridge, etc.) to the substrate being sampled beneath the deployment point.

5.2 Ponar Dredge

The Ponar dredge has side plates and a screen on the top of the sample compartment and samples a 0.05 m^2 surface area. The screen over the sample compartment permits water to pass through the sampler as it descends thus reducing turbulence around the dredge. The Ponar dredge is easily operated by one person and is one of the most effective samplers for general use on most types of substrates.

The Ponar dredge is deployed in its open configuration. It is lowered gently from the sampling platform to the substrate below the platform. After the dredge lands on the substrate, the rope is tugged upward, closing the dredge and capturing the sample. The dredge is then hauled to the surface, where it is opened to acquire the sample.

5.3 Mini-Ponar Dredge

The Mini-Ponar dredge is a smaller, much lighter version of the Ponar dredge and samples a 0.023 m^2 surface area. It is used to collect smaller sample volumes when working in industrial tanks, lagoons, ponds, and shallow water bodies. It is a good device to use when collecting sludge and sediment containing hazardous constituents because the size of the dredge makes it more amenable to field cleaning. Its use and operation are the same as described in Section 5.2, Ponar Dredge, above.

5.4 Young Grab

The Young grab sampler is a stainless steel clamshell-type grab sampler similar to a Ponar dredge. It is a clamshell-type sampler with a scissors closing action typically used for marine and estuarine sediment sampling. The Young grab sampler is one of the most consistently performing grab sampling devices for sediment sampling in both offshore marine sediments, as well as estuarine sediments. The Young sampler comes in two sizes, 0.1 m^2 and 0.04 m^2. The 0.1 m^2 is typically used when a larger volume of sediment is needed for chemistry and particle size. The 0.04 m^2 is typically used for marine benthic macroinvertebrate sampling and has become the standard grab sampler used by NOAA, USGS and USEPA.

The Young sampler is lowered to the substrate to be sampled with a cable or rope that has a catch that is released when tension is taken off the cable or rope. When the sample device is pulled up, the scissors action of the arms close the clamshell and grabs the sample.

The major difference in the Young grab sampler and other grab samplers is a square or rectangular frame attached to the device which prevents it from penetrating too deeply into soft sediments. In harder substrates, weights may be added to the frame in order to hold the grab in place to prevent collection of a "shallow" sample. A tripod frame can also be attached to the frame surrounding the Young grab sampler. The wire or rope that the grab is raised and lowered with passes through an opening in the top of the tripod and prevents the device from landing sideways or at an angle when there are strong currents or there is lateral movement of the sampling vessel during grab sampling operations.

The draw back to the Young grab sampler is that due to the weight and size of the frame, a ship with an "A" frame or a boat with a davit is required in order to raise and lower the sampler.

6 Sediment Coring Devices

6.1 General

Core samplers are used to sample vertical columns of sediment. They are particularly useful when a historical picture of sediment deposition is desired since they preserve the sequential layering of the deposit. They are also particularly useful when it is desirable to minimize the loss of material at the sediment-water interface. Many types of coring devices have been developed, depending on the depth of water from which the sample is to be obtained, the nature of the bottom material and the length of core to be collected. They vary from hand-driven push tubes to electronic vibrational core tube drivers. These methods are described below in the following sections.

Coring devices are particularly useful in pollutant monitoring because turbulence created by descent through the water is minimal, thus the fines at the sediment-water interface are only minimally disturbed; the sample is withdrawn intact, permitting the removal of only those layers of interest; core liners manufactured of glass or Teflon® can be purchased, thus reducing possible sample interferences; and the samples are easily delivered to the lab for analysis in the tube in which they were collected.

The disadvantage of coring devices is that a relatively small surface area and sample size is obtained, often necessitating repetitive sampling in order to obtain the required amount of material for analysis. Because it is believed that this disadvantage is offset by the advantages, coring devices are recommended in sampling sediments for trace organic compounds or metals analyses.

6.2 Manually Deployed Push Tubes

In shallow, wadeable waters, or for diver-collected samples, the direct use of a core liner or tube manufactured of Teflon®, plastic, or glass is recommended for the collection of sediment samples. Plastic tubes are principally used for collection of samples for physical parameters such as particle size analysis and, in some instances, are acceptable when inorganic constituents are the only parameter of concern. Their use can also be extended to deep waters when SCUBA diving equipment is utilized. Teflon® or plastic is preferred to glass since they are unbreakable, reducing the possibility of sample loss or personal injury. Stainless steel push tubes are also acceptable and provide a better cutting edge and higher strength than Teflon®. The use of glass or Teflon® tubes eliminates any possible interference due to metals contamination from core barrels, cutting heads, and retainers. The tube should be approximately 12-inches in length if only recently deposited sediments (8 inches or less) are to be sampled. Longer tubes should be used when the depth of the substrate exceeds 8 inches. Soft or semi-consolidated sediments such as mud and clays have a greater adherence to the inside of the tube and thus can be sampled with larger diameter tubes. Because coarse or unconsolidated sediments, such as sands and gravel, tend to fall out of the tube, a smaller diameter push tube is normally required to obtain a sample. In extreme cases, where sample retention in the tube is problematic, core-catchers or end caps made of Teflon® should be employed. A tube about two-inches in diameter is usually the best size. The wall thickness of the tube should be about 1/3-inch for Teflon® plastic, or glass. The inside wall may be filed down at the bottom of the tube to provide a cutting edge to facilitate entry of the liner into the substrate.

Caution should be exercised not to disturb the bottom sediments when the sample is obtained by wading in shallow water (always work facing upstream and working from downstream up). The core tube is pushed into the substrate until four inches or less of the tube is above the sediment-water interface. When sampling hard or coarse substrates, a gentle rotation of the tube while it is being pushed will facilitate greater penetration and decrease core compaction. The top of the tube is then capped to provide suction and reduce the chance of losing the sample. A Teflon® plug or end cap, or a sheet of Teflon® held in place by a rubber stopper or cork may be used. After capping, the tube is slowly extracted with the suction and adherence of the sediment keeping the sample in the tube. Before pulling the bottom part of the tube and core above the water surface, it too should be capped. An alternative to the coring device is the Shelby tube. The Shelby tube has a gravity check valve at the top of the tube where an auger handle attaches. This check valve allows air and water to escape as the tube is advanced. Once the tube is to the desired depth, the check valve will close automatically forming suction on the tube; thus, holding the sample inside.

When extensive core sampling is required, such as a cross-sectional examination of a streambed with the objective of profiling both the physical and chemical contents of the sediment, complete cores are desirable. A strong coring tube such as one made from aluminum, steel or stainless steel is needed to penetrate the sediment and underlying clay or sands. To facilitate complete core collection and retention, it is recommended that the corer (like a Shelby tube) have a check valve built into the driving head which allows water and air to escape from the cutting core, thus creating a partial vacuum, helping to hold the sediment core in the tube. The corer is attached to a standard auger extension and handle, allowing it to be corkscrewed into the sediment from a boat or while wading. The coring tube is easily detached and the intact sediment core is removed with an extraction device.

Before extracting the sediment from the coring tubes, the clear supernatant above the sediment-water interface in the core should be decanted from the tube. This is accomplished by simply turning the core tube to its side, and gently pouring the liquid out until fine sediment particles appear in the waste liquid. The loss of some of the fine sediments usually occurs with this technique.

6.3 Ogeechee Sand Pounders® and Gravity Cores

In deeper, non-wadeable water bodies, sediment cores may be collected from a bridge or a boat using different coring devices such as Ogeechee Sand Pounders®, gravity cores and vibrating coring devices. All three devices utilize a core barrel with a core liner tube system. The core liner can be removed from the core barrel and replaced with a clean core liner, as needed, after each sample. Liners are made of stainless steel, Teflon® or plastic. The type of core liner and its composition should be based on the contaminants to be evaluated.

Ogeechee Sand Pounders® and gravity cores are hand-held devices that use a standard size 2-inch diameter core barrel. The core tube and liner are interchangeable between the two units. The Ogeechee® uses a slide-hammer mechanism attached to the core head that allows the sampler to pound the core tube into the sediment. The Ogeechee® is good for sandy, more consolidated sediments. The gravity core uses a guiding fin mechanism with a built-in gravity-type check valve. The gravity core is placed in the water and released at the surface to free fall to the bottom. The fin mechanism keeps the core tube upright and free from spinning in the water column as it descends. The core tube stabs the bottom, forcing the sediment into the tube. Both coring devices are equipped with removable nose pieces on the core barrel and disposable core catchers for the liner tubes. The core catchers are designed to cap the liner tube to avoid loss of the core when retrieved from the bottom. The gravity core can be modified to attach a slide hammer mechanism, similar to the Ogeechee®, to further pound the core into the sediment further if deemed necessary.

Sediment cores collected from most hand operated coring devices can suffer from either spreading or compaction when driven into the sediment, depending on the softness of the sediment. Spreading occurs when the sediment is pushed or moved to the side during the advancement of the core tube. Compaction occurs when the sediment is being pushed downward as the core tube is advanced. Both phenomena can affect the physical integrity of the core sample. For instance, the core tube may be advanced through the sediment to a depth of 36 inches, but upon examination of the recovered core, there is only 24 inches of sediment in the core tube.

6.4 Vibratory Core Tube Drivers (Vibracore®)

Vibratory Core Tube Drivers (Vibracore®) facilitate sampling of soft or loosely consolidated, saturated sediments, with minimal compaction or spreading, using lined or unlined core tubes. It is designed for use with core tubes having nominal diameters ranging from 2-inches to 4-inches OD. The Vibracore® uses an electric motor to create vibration ranges from approximately 6,000 RPM to 8,000 RPM (100 Hz to 133 Hz) depending on the resistance afforded by the sediment; the greater the resistance, the higher the frequency. The actual vibrational displacement of the Vibracore® is on the order of a few tens of thousandths of an inch, so essentially no mixing of the sediment within the tube occurs. The vibrational energy tends to re-orient the sediment particles at the lower end of the core tube, causing them to move out of the way of the advancing wall of the core tube and into a more efficient (i.e. denser) packing. This action advances the core tube with minimal compaction of the sediment.

7 Diving

7.1 General

Sediment samples can also be obtained from large streams and open water bodies such as ponds, lakes, estuarine bodies and open ocean environments by divers. Using a variety of the above mentioned methods, divers can directly access the substrate and collect sediment samples. Depending upon the sampling methods used and the required analyses, the samples may be collected directly into the containers from the substrate or they may be returned, in bulk, to the bank or other sampling platform for processing and sample container allocation.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Surface Water Sampling**

Effective Date: December 16, 2016	**Number:** SESDPROC-201-R4

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

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SESDPROC-201-R4, *Surface Water Sampling*, replaces SESDPROC-201-R3. **General:** Corrected any typographical, grammatical, and/or editorial errors. **Title Page:** Chris Decker was omitted as an author. Updated cover page to represent SESD reorganization. John Deatrick was not listed as the Chief of the Field Services Branch	December 16, 2016
SESDPROC-201-R3, *Surface Water Sampling*, replaces SESDPROC-201-R2.	February 28, 2013
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SESDPROC-201-R0, *Surface Water Sampling,* Original Issue	February 05, 2007

TABLE OF CONTENTS

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when collecting surface water samples for field screening or laboratory analysis.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting and handling surface water samples in the field. On the occasion that SESD field personnel determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used to obtain a surface water sample, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005, Most Recent Version

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

SESD Operating Procedure for Field pH Measurement, SESDPROC-100, Most Recent Version

SESD Operating Procedure for Field Specific Conductance Measurement, SESDPROC-101, Most Recent Version

SESD Operating Procedure for Field Turbidity Measurement, SESDPROC-103, Most Recent Version

SESD Operating Procedure for Equipment Inventory and Management, SESDPROC-108, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

SESD Operating Procedure for Packaging, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

United States Environmental Protection Agency (US EPA). 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

SESD Operating Procedure for Field Sampling Quality Control, SESDPROC-011, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when collecting surface water samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines should be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when collecting surface water samples.

- Special care must be taken not to contaminate samples. This includes storing samples in a secure location to preclude conditions which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples shall conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Documentation of field sampling is done in a bound logbook.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as air bills, bills of lading, etc., shall be retained by the project leader and stored in a secure place.

2 Special Sampling Considerations

2.1 Volatile Organic Compounds (VOC) Analysis

Surface water samples for VOC analysis must be collected in 40 ml glass vials with Teflon® septa. The vial may be either preserved with concentrated hydrochloric acid or they may be unpreserved. Preserved samples have a two-week holding time, whereas, unpreserved samples have only a seven-day holding time. In the great majority of cases, the preserved vials are used to take advantage of the extended holding time. In some situations, however, it may be necessary to use the unpreserved vials. For example, if the surface water sample contains a high concentration of dissolved calcium carbonate, there may be an effervescent reaction between the hydrochloric acid and the water, producing large numbers of fine bubbles. This will render the sample unacceptable. In this case, unpreserved vials should be used and arrangements must be confirmed with the laboratory to ensure that they can accept the unpreserved vials and meet the shorter sample holding times.

The samples should be collected with as little agitation or disturbance as possible. The vial should be filled so that there is a reverse or convex meniscus at the top of the vial and absolutely no bubbles or headspace should be present in the vial after it is capped. After the cap is securely tightened, the vial should be inverted and tapped on the palm of one hand to see if any undetected bubbles are dislodged. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Samples for VOC analysis must be collected using either stainless steel or Teflon® equipment.

2.2 Special Precautions for Surface Water Sampling

- A clean pair of new, non-powdered, disposable gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come in contact with the media being sampled and should be changed any time during sample collection when their cleanliness is compromised.
- Sample containers for samples suspected of containing high concentrations of contaminants shall be stored separately.
- All background or control samples shall be collected and placed in separate ice chests or shipping containers. Sample collection activities shall proceed progressively from the least suspected contaminated area to the most suspected

contaminated area. Samples of waste or highly contaminated media must not be placed in the same ice chest as environmental (i.e., containing low contaminant levels) or background samples.

- If possible, one member of the field sampling team should take all the notes and photographs, fill out tags, etc., while the other members collect the samples.
- Samplers must use new, verified and certified-clean disposable or non-disposable equipment cleaned according to procedures contained in SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, or SESD Operating Procedure for Field Cleaning and Decontamination at the FEC, SESDPROC-206, for collection of samples for trace metals or organic compound analyses.

2.3 Sample Handling and Preservation Requirements

1. Surface water samples will typically be collected either by directly filling the container from the surface water body being sampled or by decanting the water from a collection device such as a stainless steel scoop or other device.
2. During sample collection, if transferring the sample from a collection device, make sure that the device does not come in contact with the sample containers.
3. Place the sample into appropriate, labeled containers. Samples collected for VOC analysis must not have any headspace (see Section 2.1, Volatile Organic Compounds (VOC) Analysis). All other sample containers must be filled with an allowance for ullage.
4. All samples requiring preservation must be preserved as soon as practically possible, ideally immediately at the time of sample collection. If preserved VOC vials are used, these will be preserved with concentrated hydrochloric acid by ASB personnel prior to departure for the field investigation. For all other chemical preservatives, SESD will use the appropriate chemical preservative generally stored in an individual single-use vial as described in the SESD Operating Procedure for Field Sampling Quality Control (SESDPROC-011). The adequacy of sample preservation will be checked after the addition of the preservative for all samples, except for the samples collected for VOC analysis. If it is determined that a sample is not adequately preserved, additional preservative should be added to achieve adequate preservation. Preservation requirements for surface water samples are found in the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM).
5. All samples preserved using a pH adjustment (except VOCs) must be checked, using pH strips, to ensure that they were adequately preserved. This is done by pouring a small volume of sample over the strip. Do not place the strip in the sample. Samples requiring reduced temperature storage should be placed on ice immediately.

2.4 Quality Control

If possible, a control sample should be collected from a location not affected by the possible contaminants of concern and submitted with the other samples. In streams or other bodies of moving water, the control sample should be collected upstream of the sampled area. For impounded bodies of water, particularly small lakes or ponds, it may be difficult or inappropriate to obtain an unbiased control from the same body of water from which the samples are collected. In these cases, it may be appropriate to collect a background sample from a similar impoundment located near the sampled body of water if there is a reasonable certainty that the background location has not been impacted. Equipment blanks should be collected if equipment is field cleaned and re-used on-site or if necessary to document that low-level contaminants were not introduced by pumps, bailers or other sampling equipment.

2.5 Records

Information generated or obtained by SESD personnel will be organized and accounted for in accordance with SESD records management procedures found in SESD Operating Procedure for Control of Records, SESDPROC-002. Field notes, recorded in a bound field logbook, will be generated, as well as chain-of-custody documentation in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010 and SESD Operating Procedure for Sample and Evidence Management, SESDPROC-005.

3 General Considerations

3.1 General

The surface water sampling techniques and equipment described in the following sections of this procedure are designed to minimize effects on the chemical and physical integrity of the sample. If the procedures in these sections are followed, a representative sample of the surface water should be obtained.

3.2 Equipment Selection Considerations

The physical location of the investigator when collecting a sample may dictate the equipment to be used. If surface water samples are required, direct dipping of the sample container into the stream is desirable. Collecting samples in this manner is possible when sampling from accessible locations such as stream banks or by wading or from low platforms, such as small boats or piers. Wading or streamside sampling from banks, however, may cause the re-suspension of bottom deposits and bias the sample. Wading is acceptable if the stream has a noticeable current (is not impounded), and the samples are collected while facing upstream. If the stream is too deep to wade, or if the sample must be collected from more than one water depth, or if the sample must be collected from an elevated platform (bridge, pier, etc.), supplemental sampling equipment must be used.

To collect a surface water sample from a water body or other surface water conveyance, a variety of methods can be used:

- Dipping Using Sample Container
- Scoops
- Peristaltic Pumps
- Discrete Depth Samplers
- Bailers
- Buckets
- Submersible Pumps
- Automatic Samplers

Regardless of the method used, precautions should be taken to ensure that the sample collected is representative of the water body or conveyance. These methods are discussed in the following sections.

4 Dipping Using Sample Container

A sample may be collected directly into the sample container when the surface water source is accessible by wading or other means. The sampler should face upstream if there is a current and collect the sample without disturbing the bottom sediment. The surface water sample should always be collected prior to the collection of a sediment sample at the same location. The sampler should be careful not to displace the preservative from a pre-preserved sample container, such as the 40-ml VOC vial.

5 Scoops

Stainless steel scoops provide a means of collecting surface water samples from surface water bodies that are too deep to access by wading. They have a limited reach of about eight feet and, if samples from distances too far to access using this method are needed, a mobile platform, such as a boat, may be required.

Stainless steel scoops are useful for reaching out into a body of water to collect a surface water sample. The scoop may be used directly to collect and transfer a surface water sample to the sample container, or it may be attached to an extension in order to access the selected sampling location.

6 Peristaltic Pumps

Another device that can be effectively used to sample a water column, such as a shallow pond or stream, is the peristaltic pump/vacuum jug system. The peristaltic pump can be used to collect a water sample from any depth if the pump is located at or near the surface water elevation. There is no suction limit for these applications. The use of a metal conduit to which the tubing is attached, allows for the collection of a vertical sample (to about a 25-foot depth) which is representative of the water column. The tubing intake is positioned in the water column at the desired depth by means of the conduit. Using this method, discrete samples may be collected by positioning the tubing intake at one depth or a vertical composite may be collected by moving the tubing intake at a constant rate vertically up and down the water column over the interval to be composited.

Samples for VOC analysis cannot be collected directly from the peristaltic pump discharge or from the vacuum jug. If a peristaltic pump is used for sample collection and VOC analysis is required, the VOC sample must be collected using one of the "soda straw" variations. Ideally, the tubing intake will be placed at the depth from which the sample is to be collected and the pump will be run for several minutes to fill the tubing with water representative of that interval. After several minutes, the pump is turned off and the tubing string is retrieved. The pump speed is then reduced to a slow pumping rate and the pump direction is reversed. After turning the pump back on, the sample stream is collected into the VOC vials as it is pushed from the tubing by the pump. Care must be taken to prevent any water that was in contact with the silastic pump head tubing from being incorporated into the sample.

7 Discrete Depth Samplers

When discrete samples are desired from a specific depth, and the parameters to be measured do not require a Teflon®-coated sampler, a standard Kemmerer or Van Dorn sampler may be used. The Kemmerer sampler is a brass cylinder with rubber stoppers that leave the ends of the sampler open while being lowered in a vertical position, thus allowing free passage of water through the cylinder. The Van Dorn sampler is plastic and is lowered in a horizontal position. In each case, a messenger is sent down a rope when the sampler is at the designated depth, to cause the stoppers to close the cylinder, which is then raised. Water is removed through a valve to fill respective sample containers. With a rubber tube attached to the valve, dissolved oxygen sample bottles can be properly filled by allowing an overflow of the water being collected. With multiple depth samples, care should be taken not to disturb the bottom sediment, thus biasing the sample.

When metals and organic compounds parameters are of concern, then a double-check valve, stainless steel bailer or Kemmerer sampler should be used to collect the sample.

8 Bailers

Teflon® bailers may also be used for surface water sampling if the study objectives do not necessitate a sample from a discrete interval in the water column. A closed-top bailer with a bottom check-valve is sufficient for many studies. As the bailer is lowered through the water column, water is continually displaced through the bailer until the desired depth is reached, at which point the bailer is retrieved. This technique may not be successful where strong currents are found.

9 Buckets

A plastic bucket can be used to collect samples for measurement of water quality parameters such as pH, temperature, and conductivity. Samples collected for analysis of classical water quality parameters including but not limited to ammonia, nitrate-nitrite, phosphorus, and total organic carbon may also be collected with a bucket. Typically, a bucket is used to collect a sample when the water depth is too great for wading, it is not possible to deploy a boat, or access is not possible (excessive vegetation or steep embankments) and the water column is well mixed. The water body is usually accessed from a bridge. The bucket is normally lowered by rope over the side of the bridge. Upon retrieval, the water is poured into the appropriate sample containers

Caution should be exercised whenever working from a bridge. Appropriate measures should be taken to insure the safety of sampling personnel from traffic hazards.

10 Submersible Pumps

Submersible pumps can be used to collect surface water samples directly into a sample container. The constituents of interest should be taken into consideration when choosing the type of submersible pump and tubing to be used. If trace contaminant sampling of extractable organic compounds and/or inorganic analytes will be conducted, the submersible pump and all of its components should be constructed of inert materials such as stainless steel and Teflon®. The tubing should also be constructed of Teflon®. If re-using the same pump between sample locations, the pump should be decontaminated using SESD Operating Procedure for Field Equipment Cleaning and Decontamination, (SESDPROC-205). New tubing should be used at each sample location.

If the samples will be analyzed for classical parameters such as ammonia, nitrate-nitrite, phosphorus, or total organic carbon, the pump and tubing may be constructed of components other than stainless steel and Teflon®. The same pump and tubing may be re-used at each sampling station after rinsing with deionized water and then purging several volumes of sample water through the pump and tubing prior to filling the sample containers.

Either a grab or composite sample can be collected using a submersible pump. A composite sample can be collected by raising and lowering the pump throughout the water column. If a composite sample is collected, it may be necessary to pump the sample into a compositing vessel for mixing prior to dispensing into the sample containers. If a compositing vessel is required, it should be constructed of materials compatible with the constituents of concern and decontaminated between sample stations according to appropriate procedures, again depending on the constituents of concern.

11 Automatic Samplers

Where unattended sampling is required (e.g., storm-event sampling, time-of-travel studies) an automatic sampler may be used. The automatic sampling device may be used to collect grab samples based on time, in-stream flow or water level or used to collect composite samples as dictated by the study data needs. The automatic sampling device should be calibrated prior to deployment to insure the proper volume is collected. The manufacturer's instruction manual should be consulted for automatic sampler operation.

12 Trace-Level Mercury Sampling

In order to prevent contamination during sample collection, Region 4 has developed this sampling procedure for trace-level mercury analysis (< 1 part per trillion). This procedure is based on EPA Method 1669.

A vacuum chamber assembly is utilized to collect surface water samples for trace-level mercury analyses. The vacuum chamber assembly consists of the following: 1) an airtight acrylic, cylindrical chamber with an o-ring sealed lid to hold the sample bottle, 2) a Teflon® sample tubing that connects to a centered Teflon® compression fitting on top of the chamber. The other end of the tubing passes through a rigid Teflon® pole for stability and has a modified magnetic screen holder at the intake, and a hand vacuum pump. The chamber is designed to hold a 2-liter sample bottle; however, smaller sample containers may be utilized with a spacer inserted into the chamber. A two inch square of 100 µm Nitex® screen is used on the magnetic screen holder at the intake to prevent large pieces of debris from entering the sample. The screen does not prevent the passage of particulate organic matter which is often prevalent in surface water. The vacuum chamber has a second off-center compression fitting with a 4 inch piece of Teflon® tubing inserted in the fitting. A piece of clear Tygon® tubing approximately 18-24 inches long is placed over the small piece of Teflon®. The Teflon® adds stability to the tubing and keeps it from crimping. The Tygon® is attached to the hand pump and the chamber with electrical tape. The Nitex® screen intake is inserted into the water to be sampled and a vacuum is pulled on the chamber by means of the hand vacuum pump, thus drawing a water sample into a sample container placed directly beneath the intake tubing within the chamber.

Teflon® bottles or 300-Series glass bottles with single use Teflon®-lined caps may be used for sample collection. All sample containers used for collection of trace-level mercury water samples must be pre-cleaned in a laboratory as described in EPA Method 1631. Teflon® containers should also be etched on the outside of the bottle with a unique identification number for QA purposes. All bottles for trace-level sampling must be double bagged in re-sealable bags. Water samples collected for total, inorganic, methyl or ethyl mercury analyses are pumped into appropriately cleaned bottles. Preservation should be done in a clean room laboratory that has been specifically prepared for the preparation of trace level samples (positive pressure ventilation, sticky floor mats, etc.). Preservation must occur within 48 hours of sample collection, sooner if possible. Region 4 utilizes laboratory preservation of trace-level mercury samples in order to minimize the potential for contamination, and if split samples are required, they must be split in a trace-level clean room laboratory.

The following quality assurance/quality control (QA/QC) samples are collected in conjunction with low-level mercury samples:

- bottle blanks

- equipment blanks
- air deposition blanks
- trip blanks
- duplicates and
- splits

A bottle blank is prepared in the lab with reagent-grade water to ensure the cleanliness of the bottles prior to use in the field. After decontamination of the Teflon® tubing by pumping and discarding several sample container volumes of reagent-grade water through the tubing, (using the same amount of water used for sample collection in the field) an equipment blank sample is collected into an appropriately pre-cleaned sample container. Equipment blanks are collected at the beginning of each field trip and at the end of each day. The bottle blank and the equipment blank do not go out into the field and are preserved at the end of the day with the regular field samples.

Air deposition blanks are collected to determine if airborne mercury is present at the time of sample collection. The air deposition blanks consist of a pre-cleaned mercury sample container, filled with reagent-grade water by the laboratory that prepared the containers, and is shipped with the containers to the field. The air deposition blank is uncapped using "clean hands"/ "dirty hands" procedures (see below) and set near the sampling location throughout the duration of the mercury sample collection for that particular station. Once the mercury sample is collected, the air deposition blank is recapped and handled and processed with the other mercury samples. One air deposition blank is collected each day by each field crew unless atmospheric conditions or site conditions warrant additional blanks.

Trip blanks are utilized to determine if any contaminants of interest to the study are potentially introduced to the samples during storage and transport to the laboratory. Trip blanks are prepared by the laboratory which supplies the mercury sample containers. The trip blanks consist of cleaned bottles which are filled with reagent-grade water by the laboratory and shipped with the other clean sample containers. A dark plastic bag is placed in each cooler that will hold the trace-level water samples. One trip blank is placed in each trace-level cooler of samples and returned to the laboratory with the ambient trace-level water samples. All trace-level samples should be kept in the dark until they are preserved. The trip blanks are never opened in the field. Trip blanks are preserved in the clean room.

Duplicate samples are discrete samples collected at the same site and time to measure variability of collected samples and to assess sample collection consistency. Sample splits are aliquots of a minimum 500 ml poured from a single ambient sample. They must be split in a trace-level clean room laboratory.

In order to prevent cross contamination in samples analyzed for trace-level mercury in ambient surface waters, clean sampling protocols must be employed throughout the sampling effort. For each sampling event, one sampling team member is designated as "clean hands" and one as "dirty hands" (see below). All operations involving contact with the sample bottle and transfer of the sample from the sample collection device to the sample bottle are handled by the individual designated as "clean hands." "Dirty hands" is responsible for preparation of the sampling device (except the sample container) and for all other activities that do not involve direct contact with the sample.

Prior to sample collection with the vacuum chamber assembly, the Teflon® line is cleaned at each station by rinsing with ambient water as follows: A 2-liter poly bottle is placed into the chamber and filled half full with ambient water. The bottle is swirled to rinse it and the water is discarded downstream of sampling area. The same 2-liter poly bottle can be used at each station. Additional cleaning measures are not recommended as long as the chamber assembly is only used to collect ambient surface water samples. Detergent washes and acid rinses are not conducted due to potential mercury contamination from these solutions. If applicable, samples for other analyses can be collected in a poly bottle with the vacuum chamber assembly but should be collected before the trace-level sample as an additional means of flushing the sampling line prior to collection of the trace-level samples. It is not necessary to implement the "clean hands"/ "dirty hands" method for collection of non-mercury samples, but latex or vinyl gloves should be worn when any samples are collected.

Following are procedures for cleaning the vacuum chamber tubing and collection of ancillary water quality samples, if applicable:

1. Carefully approach the sampling station from downstream and downwind if possible.

2. While wearing latex or vinyl gloves, place an uncapped 2-liter poly bottle into the chamber and secure the chamber lid by attaching the spring-loaded clamps.

3. Place a new square of 100 µm Nitex® screen in the magnetic screen holder. Place the intake beneath the surface of the water (mid-depth or six inches, whichever is less) and hold firmly in place. Care should be taken not to disturb sediment particles in very shallow waters (< 4 inches deep).

4. Squeeze the hand pump until liquid starts to fill the bottle in the chamber. When the bottle is approximately half full, release the vacuum on the chamber, remove the bottle, swirl the contents and discard the water downstream. Repeat this rinse. If ancillary water quality samples are to be collected, return the 2-liter poly bottle to the chamber and pump the required volume of water to fill the appropriate ancillary sample containers. Remove the 2-liter bottle from the chamber and cap. Fill the ancillary sample bottles upon completion of the mercury sample collection.

Water samples for trace level mercury analyses should be collected immediately after the ancillary water samples have been collected according to the following procedures:

1. "Clean hands" should put on a pair of latex or vinyl gloves, then a pair of shoulder length polyethylene gloves.

2. "Dirty hands" should put on a pair of latex or vinyl gloves, retrieve the double bagged trace level sample bottle from the cooler, and open the outer bag. "Clean hands" should open the inner bag and remove the pre-cleaned Teflon® or glass bottle.

3. "Dirty hands" should open the lid on the chamber. "Clean hands" should place the sample bottle in the chamber, remove the bottle top and place it inside the chamber with the bottle.

4. "Dirty hands" should close and secure the chamber lid and using the hand pump, fill the container. The sample container should be filled to overflowing. "Dirty hands" should then release the vacuum and open the lid on the chamber.

5. "Clean hands" should place the top on the sample bottle, remove it from the chamber and place it in the inner bag and seal the bag. "Dirty hands" should seal the outer bag and place the sample in the black bag in the dark cooler. Only coolers dedicated to storage and transport of trace-level mercury samples should be used.

U.S. Environmental Protection Agency
Region 4, Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Fish Field Sampling**	
Effective Date: February 4, 2015	**Number:** SESDPROC-512-R4

Authors

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-512-R4, *Fish Field Sampling*, replaces SESDPROC-512-R3. **Cover Page:** The Ecological Assessment Branch Chief was changed from Bill Cosgrove to the Field Services Branch Chief, John Deatrick. The FQM was changed from Liza Montalvo to Hunter Johnson. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history.	February 4, 2015
SESDPROC-512-R3, *Fish Field Sampling*, replaces SESDPROC-512-R2.	April 14, 2011
SESDPROC-512-R2, *Fish Field Sampling*, replaces SESDPROC-512-R1.	December 18, 2009
SESDPROC-512-R1, *Fish Field Sampling*, replaces SESDPROC-512-R0.	November 1,2007
SESDPROC-512-R0, *Fish Field Sampling*, Original Issue	February 05, 2007

TABLE OF CONTENTS

1 General Information

1.1 Purpose

This document provides general procedures, methods, and considerations to be used and observed while collecting fish in freshwater, marine and estuarine environments.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when collecting fish in freshwater, marine and estuarine environments. Upon the realization that any of the procedures described in this document cannot be used other methods may be implemented to ensure collection. Any other procedure or methods of collection used that are not described in this document must be documented in the field log book and subsequent investigation report, along with circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and has been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Logbooks, SESDPROC-010, Most Recent Version

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version

Title 49 Code of Federal Regulations, Pts. 171 to 179, Most Recent Version

United States Environmental Protection Agency (USEPA). 1999. Rapid Bioassessment Protocols for use in Streams and Wadeable Rivers: Periphyton, Benthic Macroinvertebrates, and Fish. Second Edition. EPA 841-B-99-002. Office of Water, Washington, DC.

USEPA. 2000. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories, Third Edition. EPA 823-B-00-007. Office of Science and Technology, Office of Water, Washington, DC.

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

1.5 Taxonomic References

Boschung, H. T. and R. L., Mayden. 2004. Fishes of Alabama. Smithsonian Books, Birmingham, AL.

Marcy, B. C., D. E. Fletcher, F. D. Martin, M. H. Paller, M. J. M. Reichert. 2005. Fishes of the Middle Savannah River Basin with Emphasis on the Savannah River Site. University of Georgia Press. Athens, GA.

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Murphy, B. R. and D. W. Willis. 1996. Fisheries Techniques. American Fisheries Society. Bethesda, MD.

Jenkins, R. E. and N. M. Burkhead. 1993. Freshwater Fishes of Virginia. American Fisheries. Society, Bethesda, MD.

Page, L. M. and B. M. Burr. 1991. A Field Guide to Freshwater Fishes. Houghton Mifflin Co., Boston, MA.

1.6 General Precautions

1.6.1 Safety

All members of the sampling crew should be able to swim and be certified in Cardiopulmonary resuscitation (CPR) as well as first aid. Depending on the Data Quality Objectives (DQO) certain samples may be frozen or preserved with ethanol or formalin. Proper safety precautions must be observed when working with either preservative. Refer to the Science and Ecosystem Support Division's (SESD) Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. When using this procedure, minimize exposure to potential health hazards through the use of protective clothing, eye wear and gloves.

1.6.2 Procedural Precautions

The following precautions should be considered when performing fish collections:

- Care in handling of organisms during collection is necessary to ensure specimens are not being contaminated. This includes handling fish with latex gloves and storing fish in a sealed clean plastic bag on wet ice for no more than forty-eight hours before filleting.
- Care should be taken not to damage fish, particularly when collecting fish for use in IBI (Index of Biotic Integrity) determinations, to ensure proper taxonomic identification.
- Sample specimens that are sacrificed and subject to identification at a later date should be properly fixated with formalin and preserved in ethanol.
- Collected samples for tissue analysis should be stored in a secure location to preclude conditions, such as desiccation, which could alter the properties of the sample. Samples shall be custody sealed during long-term storage or shipment.
- Collected samples are in the custody of the sampler or sample custodian until the samples are relinquished to another party.
- If samples are transported by the sampler, they will remain under his/her custody or be secured until they are relinquished.
- Shipped samples will conform to all U.S. Department of Transportation (DOT) rules of shipment found in Title 49 of the Code of Federal Regulations (49 CFR parts 171 to 179), and/or International Air Transportation Association (IATA) hazardous materials shipping requirements found in the current edition of IATA's Dangerous Goods Regulations.
- Chain-of-custody documents shall be filled out and remain with the samples until custody is relinquished.
- All shipping documents, such as bills of lading, will be retained by the project leader and stored in a secure place.

2 Sampling Considerations

2.1 Summary of Procedure

The use of collection techniques described below should provide fish that are suitable for use in ecological assessments, tissue analyses and other relevant studies. The following procedures for fish collection are designed to minimize effects on the chemical and physical integrity of the sample. The following techniques are to be used as general guidelines and may be used in conjunction with one another as sample locations and DQO's may differ at each location. In all cases proper collection permits will be obtained from the issuing State or agency.

2.2 Gear Selection

Before selection of sampling gear, consideration should be given to biological and environmental factors that influence gear efficiency. Characteristics of the fish community being sampled are among the biological factors affecting efficiency. Susceptibility to gear varies among fish species due to differences in morphology, physiology, and behavior. Environmental factors to be considered are the size and depth of the water body to be sampled along with previous and current weather conditions, water temperature, and conductivity. General guidelines for the use of active (moving gear or nets to capture fish) and passive (fish move into stationary collection device) collection gears for fishes are found in sections 3 and 4.

2.3 Quality Control

Assure that samples are properly labelled and preserved. Fish collected for tissue analysis may be stored in clear plastic bags in coolers on wet ice for no more than forty-eight hours. Fish from multiple collection sites may be stored in the same cooler as long as each sample is stored in a clean separate plastic bag. In studies were fish length and weight are measured, length should be measured in millimeters (mm), weight in grams (g) and recorded in logbook.

A library of basic taxonomic literature is essential in aiding in the identification of specimens. The taxonomic publications in common use should be stored in the fish processing lab. These references are listed in the references section in this procedure. In addition, voucher specimens should be retained for verification from additional sources, if necessary.

2.4 Records

Records generated will include field notes, recorded in a bound waterproof logbook (in accordance with SESD Operating Procedure for Logbooks, SESDPROC-010), field data sheets for physical characterizations, digital photographs, custody tags, completed chain-of-custody forms, lab bench sheets and, if needed, completed receipt for sample forms.

3 Active Collection Methods

3.1 Electrofishing

Electrofishing is a form of active sampling with the use of electricity. It allows the user to collect fish in different types of water bodies dependent on the specific gear type chosen i.e. backpack, barge, or boat electrofishers. Before selection of electrofishing gear, consideration should be given to biological and environmental factors that influence gear efficiency.

Characteristics of fish community being sampled are among the biological factors affecting efficiency. Susceptibility to electrofishing varies among fish species due to differences in morphology, physiology, and behavior. Typically large fish are more vulnerable than small fish, bony fish are more susceptible than cartilaginous fish and fish with smaller vestigial scales (e.g., ictalurids) are more susceptible than those with large scales such as cyprinids.

WATER CONDUCTIVITY is the most IMPORTANT variable. When conductivity is low it is harder to pass electricity between the electrodes. When conductivity is high (higher than the fish tissue) current passes around the fish and they are not affected. Ideal conditions are met when the conductivity is high, but lower than the conductivity of the fish tissue. Water temperature affects fish activity and conductivity as both activity and conductivity increase with an increase in temperature. Turbidity can also affect efficiency as fish are not as easily seen. Other environmental factors that should be taken into consideration when selecting a gear type are the size and depth of the water body.

While conducting electrofishing polarized sunglasses should be worn. They allow the user to see into the water better and thus increase efficiency. Also, depending on conditions, while wading in shallow streams they allow to user to identify potential hazards and habitat structure.

Above all else consider the SAFETY for YOU and your TEAM. ELECTRIC CURRENT IS LETHAL. The best safety precautions are those that minimize contact with charged water and decrease the probability of electrocution and drowning. People conducting electrofishing surveys should wear a PFD (personal floatation device) at all times when in a boat or wading in deep or swift water, wear rubber boots or waders, wear rubber gloves and be certified in CPR.

3.1.1 Boat Electrofisher

Generally, boat electrofishers are used when the water body is larger or too deep to be wadeable. Typically reservoirs, lakes, and larger rivers require the use of a boat electrofisher. Boat size can also affect your capability to access an area or waterbody. Larger boats work well in larger water bodies such as Clarks Hill and Russell Reservoirs as their maneuverability is not as restricted. Smaller boats 14' or less are generally better suited for smaller ponds and rivers.

Boat setup:

- Gas powered AC generator used as power source
- Control box allows manipulation of current (AC vs. DC), output voltage and pulsing.
- Positive electrodes (anode) at the bow of boat, negative electrode (cathode) usually the boats hull or a cable hanging over side.

3.1.2 Barge Electrofisher

Barge electrofishers are typically used in streams or rivers when the water body is wadeable but not accessible by boat. The use of a barge electrofisher requires a minimum of 3 people. One person is in charge of the barge and monitoring the output and SAFETY of the others while the other 2 people actively electrofish. Ideally another 2 or more individuals would help by netting.

Barge setup:

- Gas powered AC generator used as power source
- Control box allows manipulation of current (AC vs. DC), output voltage and pulsing.
- Positive electrodes (anode) handheld pole with ring, negative electrode (cathode) usually a small 12” x 12” metal plate under the bow or cable hanging over side.

3.1.3 Backpack Electrofisher

Backpack electrofishers are typically used in smaller wadeable rivers and streams. They can be utilized with other backpack shockers, block nets and seines to increase efficiency. A minimum of two people is required to operate a backpack electrofisher. One person will wear the backpack electrofisher and the other will net. Optimally one should block off the ends of the reach to minimize escapement and sample moving upstream to minimize effects of turbidity.

Backpack setup:

- 24 volt large or small capacity battery.
- Backpack unit is the control box DC current only.
- Positive electrodes (anode) handheld pole with ring, negative electrode (cathode) usually a cable hanging off the rear of the backpack electrofisher.

3.1.4 Other Active Gears

Other active gears used to capture fish include trawls, dredges, surrounding nets (i.e. purse seines), cast nets and angling. Each of these gears will have an ideal set of circumstances in which they are effective. Trawls, dredges, and surrounding nets are typically used in open water for pelagic species. Cast nets can be used at depths up to 15 feet and can be efficient at collecting other species such as shrimp. Angling can be used as a general sampling method or to supplement catch from another gear and may be used in a wide variety of situations.

4 Passive Collection Methods

4.1 Passive Techniques

Passive techniques do not involve the movement of the sampling gear to collect fish. These gears are typically anchored and stationary allowing fish to move into them. Passive techniques can be grouped by their method of capture: entanglement, entrapment, and angling. Entanglement gear captures fish by ensnaring or tangling (i.e. gill and trammel nets). Entrapment gear captures organisms as they enter an enclosed area and are unable to escape. Entrapment devices include hoop nets, fyke nets, slat traps, and crap pots. Angling methods involve setting an individual or line of stationary hooks that are baited such as a trotline. When selecting a passive gear for use, selectivity of the gear must be considered based on the target species.

4.2 Entanglement Gear

Gill and trammel nets are considered entanglement gears that capture fish by ensnaring or tangling. When selecting these gears careful consideration should be given to gear selectivity. These gears can be set at the surface or submerged.

4.2.1 Gill Nets

Gill nets consist of a vertical panel(s) of mesh with a float line on top and lead weights on bottom. The size of the mesh can vary and is expressed in bar or stretch measure. Nets that have more than one panel of different size mesh are experimental gill nets and can capture a wide variety of size classes. Typically gill nets are set in shallow waters on the surface or submerged up to 50 meters. When choosing a site to set gill nets consideration should be given to target species and submerged obstructions such as trees that may interfere with collection or retrieval of nets.

4.2.2 Trammel Nets

Trammel nets are typically constructed of three parallel mesh panels of netting suspended from a float line with a lead line on bottom. The outer panels consist of a larger mesh while the inner panel mesh is smaller and hangs longer and lose compared to the outer panels. Fish will pass through the outer panel into the middle in which they push through the second outer panel thus, capturing themselves in a pocket of mesh. Trammel nets are generally used for larger fish in shallow lakes or reservoir and can also be fished in rivers by letting them drift with the current. If drifting trammel nets, they should be carefully watched in order to avoid obstructions and retrieve fish upon immediate capture.

4.3 Entrapment Gear

Common entrapment devises are: hoop nets, fyke nets and traps (i.e. slat traps and crab pots). Entrapment devises are designed to capture aquatic organisms as they enter the gear through their own movement. Generally, organisms may be attracted to the entrapment gear by baiting it or the gears ability to create cover. Knowledge of your target species is important when selecting a type of entrapment gear.

4.3.1 Hoop & Fyke Nets

Hoop and fyke nets are constructed of wooden, steel, plastic, or fibreglass hoops which are connected by netting to for a cylindrical net with funnel shaped entrances between each hoop. Hoop net are generally designed for use in riverine habitats by staking the entrance out upstream and allowing the current to hold the net open. Fyke nets are designed for use in lakes or reservoirs as they also incorporate a panel or wing that protrudes from the mouth of the net that acts as a barrier to funnel fish into the net. Hoop nets can also be utilized in ponds and lakes and can be baited.

4.3.2 Traps

Generally, traps are small, portable, and rigid with an opening that allows for the target species to enter. They are generally designed to capture bottom dwelling fish, crustaceans, and species that are fond of tight cover (i.e. catfish). Traps are usually connected to a surface float or can be tethered to shore and are typically baited.

4.3.3 Angling

Angling with hook and line can also be an effective way to collect fish samples depending on the target species. Trotlines and limb lines are among the passive techniques that can be used as the primary method for capture or to supplement others. Trotlines are typically a long line with a multitude of baited hooks. The trotline is anchored at both ends and set in the water for a period of time. Limb lines are baited hooks that are attached to tree limbs or stakes on the shore. Both these methods can be used in rivers, lakes, and reservoirs.

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Region 4 U.S. Environmental Protection Agency Science and Ecosystem Support Division Athens, Georgia	
OPERATING PROCEDURE	
Title: **Sample and Evidence Management**	
Effective Date: January 29, 2013	**Number:** SESDPROC-005-R2
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Revision History

This table shows changes to this controlled document over time. The most recent version is presented in the top row of the table. Previous versions of the document are maintained by the SESD Document Control Coordinator.

History	Effective Date
SESDPROC-005-R2, *Sample and Evidence Management*, replaces SESDPROC-005-R1 **General:** Corrected any typographical, grammatical, and/or editorial errors. **General:** Replaced all references to FORMS with the generic term sample custody software program. The specific sections are listed below: Section 3.3 Section 3.4 Section 4.2 Section 5 **Title Page:** Changed the EIB Branch Chief from Antonio Quinones to Danny France. Changed the EAB Branch Chief from Bill Cosgrove to John Deatrick (Acting). Changed the Field Quality Manager from Laura Ackerman to Bobby Lewis. **Revision History:** In the last sentence, changed Field Quality Manager to Document Control Coordinator. **Section 1.2:** Added the following statement - Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use. **Section 1.3:** Changed requirement so that the DCC is responsible for ensuring the most recent version of the procedure is placed on the SESD LAN and for maintaining records of review conducted prior to its issuance. Deleted reference to the H: drive. Section 2.2 Changed requirement for comments on sample label to make comments optional. Section 2.2.2 Removed requirement for printed copies of photographs in the official file. Section 3.3 Removed different custody requirements for criminal investigations so that all projects are treated consistently. Section 3.4 Removed statement in the fourth paragraph regarding retention of paper air bills in the official project file to reduce unnecessary paperwork. The air bill number or shipment tracking number is recorded on the chain of custody.	January 29, 2013

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SESDPROC-005-R1, *Sample and Evidence Management*, replaces SESDPROC-005-R0 **General** Updated referenced procedures to reflect most recent version. Replaced "shall" with "will". **Cover Page:** Changed title for Antonio Quinones from Environmental Investigation Branch to Enforcement and Investigations Branch. Changed Bill Cosgrove's title from Acting Chief to Chief. **Section 1.3** Updated information to reflect that procedure is located on the H: drive of the LAN. **Section 1.4** Added reference for the SESD Operating Procedure for Control of Records. Alphabetized and revised the referencing style for consistency. **Section 2.2.3** Added that Confidential Business Information will be handled in accordance with SESD Operating Procedure for Control of Records.	November 1, 2007
SESDPROC-005-R0, Sample and Evidence Management, Original Issue	February 05, 2007

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TABLE OF CONTENTS

1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when handling and managing samples and other types of evidence after their collection and during delivery to the laboratory.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when handling and managing samples and other evidence collected to support SESD field investigations. On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that another procedure must be used, the variant procedure will be documented in the field log book, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, SESDPROC-209, Most Recent Version

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Digital Camera Guidance for EPA Civil Inspections and Investigations, July 2006

2 Sample and Evidence Identification

2.1 Introduction

Sample identification, chain-of-custody records, receipt for sample records and other field records will be legibly recorded with waterproof, non-erasable ink, unless otherwise specified. If errors are made in any of these documents, corrections will be made by crossing a single line through the error and entering the correct information. All corrections must be initialed and dated. If possible, all corrections should be made by the individual making the error.

Following are definitions of terms used in this section:

Field Investigator
Any individual who performs or conducts field sampling, observation and/or measurement activities in support of field investigations

Project Leader
The individual with overall responsibility for conducting a specific field investigation in accordance with this procedure

Field Sample Custodian
Individual responsible for identifying the sample containers and maintaining custody of the samples and the Chain-of-Custody Record

Sample Team Leader
An individual designated by the project leader to be present during and responsible for all activities related to the collection of samples by a specific sampling team

Sampler
The individual responsible for the actual collection of a sample

Transferee
Any individual who receives custody of samples subsequent to release by the field sample custodian

Laboratory Sample Custodian
Individual responsible for accepting custody of samples from the field sample custodian or a transferee

One individual may fulfill more than one of the roles described above.

2.2 Sample and Evidence Identification Procedures

2.2.1 Sample Identification

The method of sample identification used depends on the type of sample collected. Field measurement samples are those collected for specific field analysis or measurement where the data are recorded directly in bound field logbooks or on the Chain-of-Custody Record. Examples of field measurements and analyses include XRF, pH, temperature, dissolved oxygen and conductivity. Samples collected for laboratory analysis will be identified by using a stick-on label or a tag which is attached to the sample container. In some cases such as biological samples, the label or tag may have to be affixed to a bag containing the sample. If a sample tag is used, the sample should be placed in a bag, then the sample and the tag will be placed in a second bag.

The following information will be included on the sample label or tag using waterproof, non-erasable ink:

- Project number;
- Field identification or sample station number;
- Date and time of sample collection;
- Designation of the sample as a grab or composite;
- Whether the sample is preserved or unpreserved;
- The general types of analyses to be performed.

Other information such as readily detectable or identifiable odor, color, or known toxic properties may be added as deemed necessary by the project leader or sample custodian.

2.2.2 Photograph, Digital Still Image and Video Identification

<u>Photographs and Digital Still Images</u>

When photographs or digital images are taken for purposes of documenting and supporting a field investigation, a record of each exposure or image will be kept in a bound field logbook. The following information will be recorded in the logbook:

- An accurate description of what the photograph or image shows, including orientation, if appropriate;

- The date and time that the photograph or image was taken;

- The name of the individual who took the photograph or digital image.

When photographs are taken with a film camera, the film should be developed with the negatives supplied uncut, if possible. The identifying information that was recorded in the field logbook will be entered on the back of the prints.

When digital images are obtained during a field investigation, an electronic copy of the unaltered investigation-related images will be placed in the official files. If deemed necessary due to project requirements, a printed copy of the original photographs may be placed in the official file. For enforcement cases, it is imperative that the individual who took the image be identified in the field logbook in the event their testimony is required.

Video

When a video recording is used as evidence in an enforcement case, the following information should be recorded in a bound field logbook:

- The date and time that the video was recorded;

- A brief description of the subject of the video;

- The person recording the video.

An audio record may also be included in the video tape with the above logistical information, as well as a narrated description of the video record.

A label will be placed on the video media with the appropriate identifying information (i.e., project name, project number, date, location etc.). In the event testimony regarding a video recording is required for an enforcement case, one individual should be responsible for recording the video for each case. The original, unaltered recording will be placed in the official files.

2.2.3 Identification of Physical Evidence

Physical evidence, other than samples, will be identified, when possible, by recording the necessary information on the evidence. When samples are collected from vessels or containers which can be moved (drums for example), the vessel or container should be marked with the field identification or sample station number for future identification. The vessel or container may be labeled with an indelible marker (e.g., paint stick or spray paint). The vessel or container need not be marked if it already has a unique marking; however, these markings will be recorded in the bound field logbooks. In addition, it is suggested that photographs of any physical evidence (markings, etc.) be taken and the necessary information recorded in the field logbook.

Occasionally, it is necessary to obtain copies of recorder and/or instrument charts from facility owned analytical equipment, flow recorders, etc., during field investigations and inspections. A unique identifier will be recorded on the document with that information as well as the following recorded in the logbook:

- Starting and ending time(s) and date(s) for the chart;
- An instantaneous measurement of the media being measured by the recorder will be taken and entered at the appropriate location on the chart along with the date and time of the measurement; and
- A description of the location being monitored and other information required to interpret the data such as type of flow device, chart units, factors, etc.

The field investigator will indicate who the chart (or copy of the chart) was received from and enter the date and time, as well as the field investigator's initials.

Documents such as technical reports, laboratory reports, etc., should be marked with the field investigator's signature, the date, the number of pages and from whom they were received. Documents that are claimed by a facility to be "confidential" and, therefore, potentially subject to the Confidential Business Information requirements, will be handled in accordance with SESD Operating Procedure for Control of Records (SESDPROC-002).

3 Chain-of-Custody Procedures

3.1 Introduction

Chain-of-custody procedures are comprised of the following elements: 1) maintaining custody of samples or other evidence, and 2) documentation of the chain-of-custody for evidence. To document chain-of-custody, an accurate record must be maintained to trace the possession of each sample, or other evidence, from the moment of collection to its introduction into evidence.

3.2 Sample Custody

A sample or other physical evidence is in custody if:

- It is in the actual possession of an investigator;
- It is in the view of an investigator, after being in their physical possession;
- It was in the physical possession of an investigator and then they secured it to prevent tampering; and/or
- It is placed in a designated secure area.

3.3 Documentation of Chain-of-Custody

The following are used to identify and demonstrate how sample integrity is maintained and custody is ensured.

Sample Identification
A stick-on sample label or a tag should be completed for each sample container using waterproof, non-erasable ink as specified in Section 2.2.1.

Sample Seals
If appropriate, samples should be sealed as soon as possible following collection using a custody seal with EPA identification. The sample custodian or project leader will write the date and their initials on the seal. The use of custody seals may be waived if field investigators keep the samples in their custody as defined in Section 3.2, from the time of collection until the samples are delivered to the laboratory analyzing the samples.

Field Sample Custodian
The field sample custodian is the person designated by the project leader to receive and manage custody of samples while in the field, including labeling and custody sealing.

Chain-of-Custody Record

The field Chain-Of-Custody record is used to document the custody of all samples or other physical evidence collected and maintained by investigators. All physical evidence or samples will be accompanied by a Chain-Of-Custody Record. This form may be generated by sample custody management software (Section 5) or it may be a pre-printed multi-sheet carbonless form for hand entry of required information. The Chain-Of-Custody Record documents transfer of custody of samples from the sample custodian to another person, to the laboratory or other organizational elements. The Chain-of-Custody Record will not be used to document the collection of split samples where there is a legal requirement to provide a receipt for samples (see Section 4, Receipt for Samples Form (CERCLA/RCRA/TSCA)). The Chain-Of-Custody Record also serves as a sample logging mechanism for the laboratory sample custodian. A separate Chain-of-Custody Record should be used for each final destination or laboratory used during the investigation.

All information necessary to fully and completely document the sample collection and required analyses must be recorded in the appropriate spaces to complete the field Chain-Of-Custody Record. The following requirements apply to Chain-Of-Custody records generated by either sample custody management software or by hand entry on pre-printed forms:

- All sampling team leaders must sign in the designated signature block.

- One sample should be entered on each line and not be split among multiple lines.

- If multiple sampling teams are collecting samples, the sampling team leader's name should be clearly indicated for each sample.

- The total number of sample containers for each sample must be listed in the appropriate column. Required analyses should be entered in the appropriate location on the Chain-of-Custody Record.

- The field sample custodian, project leader or other designee, and subsequent transferee(s) should document the transfer of the samples listed on the Chain-of-Custody Record. Both the person relinquishing the samples and the person receiving them must sign the form. The date and time that this occurs should be documented in the proper space on the Chain-of-Custody Record. The exception to this requirement would be when packaged samples are shipped with a common carrier. Even though the common carrier accepts the samples for shipment, they do not sign the Chain-of-Custody Record as having received the samples.

- The last person receiving the samples or evidence will be the laboratory sample custodian or their designee(s).

The Chain-of-Custody Record is a uniquely identified document. Once the Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated based upon its inclusion of all of the above information in a legible format.

If chain-of-custody is required for documents received during investigations, the documents should be placed in large envelopes, and the contents should be noted on the envelope. The envelope will be sealed and an EPA custody seal placed on the envelope such that it cannot be opened without breaking the seal. A Chain-Of-Custody Record will be maintained for the envelope. Any time the EPA seal is broken, that fact will be noted on the Chain-Of-Custody Record and a new seal affixed, as previously described in this section.

Physical evidence such as video tapes or other small items will be placed in an evidence bag or envelope and an EPA custody seal should be affixed so that they cannot be opened without breaking the seal. A Chain-Of-Custody Record will be maintained for these items. Any time the EPA seal is broken, that fact will be noted on the Chain-of-Custody Record and a new seal affixed.

EPA custody seals can be used to maintain custody of other items when necessary by using similar procedures as those previously outlined in this section.

Samples should not be accepted from other sources unless the sample collection procedures used are known to be acceptable, can be documented and the sample chain-of-custody can be established. If such samples are accepted, a standard sample label containing all relevant information and the Chain-Of-Custody Record will be completed for each set of samples.

3.4 Transfer of Custody with Shipment

Transfer of custody is accomplished by the following:

- Samples will be properly packaged for shipment in accordance with the procedures outlined in SESD Operating Procedure for Packing, Marking, Labeling and Shipping of Environmental and Waste Samples (SESDPROC-209).

- All samples will be accompanied by the laboratory copy of the Chain-Of-Custody Record. If pre-printed forms are used, the white and pink sheets will be sent. If sample custody management software is used to generate the Chain-Of-Custody Record, the laboratory copy is identified with an “L” in the upper right corner. If multiple coolers are needed for shipment to a particular laboratory, the laboratory copy of the Chain-Of-Custody Record for the entire shipment is placed in a sealed plastic bag in one of the coolers. When shipping samples via common carrier, the "Relinquished By" box should be filled in; however, the "Received By" box should be left blank. The laboratory sample custodian is responsible for receiving custody of the samples and will fill in the "Received By" section of the Chain-of-

Custody Record. One copy of the Record will be provided to and retained by the project leader. After samples have been received and accepted by the laboratory, a copy of the Chain-of-Custody Record, with ASB sample identification numbers, will be transmitted to the project leader. This copy will become a part of the project file.

- If sent by mail, the package will be registered with return receipt requested. If sent by common carrier, an Air Bill should be used. The Air Bill number, shipment tracking number or registered mail serial number will be recorded in the remarks section of the Chain-Of-Custody Record.

4 Receipt for Samples Form (CERCLA/RCRA/TSCA)

4.1 Introduction

Section 3007 of the Resource Conservation and Recovery Act (RCRA) of 1976 and Section 104 of the Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA or Superfund) of 1980 require that a "receipt" for all facility samples collected during inspections and investigations be given to the owner/operator of each facility before the field investigator departs the premises. The Toxic Substances Control Act (TSCA) contains similar provisions. The laws do not require that homeowners or other off-site property owners be given this form.

4.2 Receipt for Samples Form

If necessary, a Receipt for Samples form, using either the pre-printed form or one generated by sample custody management software, is to be used to satisfy the receipt for samples provisions of RCRA, CERCLA and TSCA. The form also documents that split samples were offered and either "Received" or "Declined" by the owner/operator of the facility or site being investigated (if a sample is split with a facility, state regulatory agency or other party representative, the recipient should be provided (if enough sample is available) with an equal weight or volume of sample). All information must be supplied in the indicated spaces to complete the Receipt for Samples form.

- The sampler(s) must sign the form in the indicated location

- Each sample collected from the facility or site must be documented in the sample record portion of the form. The sample station number, date and time of sample collection, composite or grab sample designation, whether or not split samples were collected (yes or no should be entered under the split sample column), a brief description of each sampling location and the total number of sample containers for each sample must be entered.

- The bottom of the form is used to document the site operator's acceptance or rejection of split samples. The project leader must sign and complete the information in the "Split Samples Transferred By" section (date and time must be entered). If split samples were not collected, the project leader should initial and place a single line through "Split Samples Transferred By" in this section. The operator of the site must indicate whether split samples were received or declined and sign the form. The operator must give their title, telephone number and the date and time they signed the form. If the operator refuses to sign the form, the sampler(s) should note this fact in the operator's signature block and initial this entry.

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The Receipt for Samples form is an accountable document after it is completed. A copy of the form is to be given to the facility or site owner/operator. The original form must be maintained in the project files.

5 Sample Custody Management Software

The container labels and the Chain-of-Custody record should be generated using a sample custody management software to streamline the documentation required by SESD and/or the Contract Laboratory Program (CLP) for sample identification and chain-of-custody. When possible, the sample custody management software should be used during all field investigations. Once the appropriate information is entered into the computer, the software will generate stick-on labels for the sample containers and will generate sample receipt forms and chain-of-custody records for the appropriate laboratory. The advantages to this system include faster processing of samples and increased accuracy. Accuracy is increased because the information is entered only once, and consequently, consistent for the bottle labels, sample receipt forms and chain-of-custody records. .

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Region 4 **U.S. Environmental Protection Agency** **Science and Ecosystem Support Division** **Athens, Georgia**	
OPERATING PROCEDURE	
Title: **Packing, Marking, Labeling and Shipping of Environmental and Waste Samples**	
Effective Date: February 4, 2015	**Number:** SESDPROC-209-R3
Authors	
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SESDPROC-209-R3, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R2. **Cover Page:** Changes made to reflect reorganization of SESD from two field branches to one: John Deatrick listed as the Chief, Field Services Branch. The FQM was changed from Liza Montalvo to Hunter Johnson. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history.	February 4, 2015
SESDPROC-209-R2, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R1.	April 20, 2011
SESDPROC-209-R1, *Packing, Marking, Labeling and Shipping of Environmental and Waste Samples*, replaces SESDPROC-209-R0.	November 1, 2007
SESDPROC-209-R0, Packing, Marking, Labeling and Shipping of Environmental and Waste Samples, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

Regulations for packing, marking, labeling, and shipping of dangerous goods by air transport are promulgated by Department of Transportation under 49 CFR, Subchapter C, Hazardous Materials Regulations, and the International Air Transport Authority (IATA), which is equivalent to United Nations International Civil Aviation Organization (UN/ICAO). Transportation of hazardous materials (dangerous goods) by EPA personnel is covered by EPA Order 1000. This document describes general and specific procedures, methods and considerations to be used and observed by SESD field investigators when packing, marking, labeling and shipping environmental and waste samples to ensure that all shipments are in compliance with the above regulations and guidance.

1.2 Scope/Application

The procedures contained in this document are to be used by field personnel when packing, marking, labeling, and shipping environmental samples and dangerous goods by air transport. Samples collected during field investigations or in response to a hazardous materials incident must be classified prior to shipment, as either environmental or hazardous materials (dangerous goods) samples.

In general, environmental samples include drinking water, most groundwater and ambient surface water, soil, sediment, treated municipal and industrial wastewater effluent, biological specimens, or any samples not expected to be contaminated with high levels of hazardous materials. Samples collected from process wastewater streams, drums, bulk storage tanks, soil, sediment, or water samples from areas suspected of being highly contaminated may require shipment as dangerous goods.

Government employees transporting samples or hazardous materials (i.e., preservatives or waste samples) in government vehicles are not subject to the requirements of this section in accordance with 49 CFR 171.1(d)(5). EPA contractors, however, are not covered by this exemption and may not transport these materials without full compliance with 49 CFR.

Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

International Air Transport Authority (IATA). Dangerous Goods Regulations, Most Recent Version.

Title 40 Code of Federal Regulations (CFR), Pt. 136.3, Identification of Test Procedures, July 1, 2001. See Table II, Footnote 3.

Title 49 CFR, Pt. 171.1(d)(5), Applicability of Hazardous Materials Regulations (HMR) to Persons and Functions.

United States Department of Transportation (US DOT). 2003. Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No. 02-0093, February 13, 2003.

US Environmental Protection Agency (US EPA) Order 1000.18, February 16, 1979.

US EPA. 1981. "Final Regulation Package for Compliance with DOT Regulations in the Shipment of Environmental Laboratory Samples," Memo from David Weitzman, Work Group Chairman, Office of Occupational Health and Safety (PM-273), April 13, 1981.

US EPA. 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA.

US EPA. Analytical Support Branch Laboratory Operations and Quality Assurance Manual. Region 4 SESD, Athens, GA, Most Recent Version.

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version.

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when packing, marking, labeling, and shipping environmental or waste samples. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional.

2 Shipment of Dangerous Goods

The project leader is responsible for determining if samples collected during a specific field investigation meet the definitions for dangerous goods. If a sample is collected of a material that is listed in the Dangerous Goods List, Section 4.2, IATA, then that sample must be identified, packaged, marked, labeled, and shipped according to the instructions given for that material. If the composition of the collected sample(s) is unknown, and the project leader knows or suspects that it is a regulated material (dangerous goods), the sample may not be offered for air transport. If the composition and properties of the waste sample or highly contaminated soil, sediment, or water sample are unknown, or only partially known, the sample may not be offered for air transport.

In addition, the shipment of pre-preserved sample containers or bottles of preservatives (e.g., NaOH pellets, HCL, etc.) which are designated as dangerous goods by IATA is regulated. Shipment of nitric acid is strictly regulated. Consult the IATA Dangerous Goods Regulations for guidance. ***Dangerous goods must not be offered for air transport by any personnel except SESD's dangerous goods shipment designee or other personnel trained and certified by IATA in dangerous goods shipment.***

3 Shipment of Environmental Samples

Guidance for the shipment of environmental laboratory samples by personnel is provided in a memorandum dated March 6, 1981, subject "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Laboratory Samples". By this memorandum, the shipment of the following unpreserved samples is not regulated:

- Drinking water
- Treated effluent
- Biological specimens
- Sediment
- Water treatment plant sludge
- POTW sludge

In addition, the shipment of the following preserved samples is not regulated, provided the amount of preservative used does not exceed the amounts found in 40 CFR 136.3 or the USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version. This provision is also discussed in correspondence between DOT and EPA (Department of Transportation, Letter from Edward T. Mazzullo, Director, Office of Hazardous Materials Standards, to Henry L. Longest II, Acting Assistant Administrator, USEPA, Ref No.: 02-0093, February 13, 2003). It is the shippers' (individual signing the air waybill) responsibility to ensure that proper amounts of preservative are used:

- Drinking water
- Ambient water
- Treated effluent
- Biological specimens
- Sediment
- Wastewater treatment plant sludge
- Water treatment plant sludge

Samples determined by the project leader to be in these categories are to be shipped using the following protocol, developed jointly between USEPA, OSHA, and DOT. This procedure is documented in the "Final National Guidance Package for Compliance with Department of Transportation Regulations in the Shipment of Environmental Laboratory Samples."

Untreated wastewater and sludge from Publicly Owned Treatment Works (POTWs) are considered to be "diagnostic specimens" (not environmental laboratory samples). However, because they are not considered to be etiologic agents (infectious) they are not restricted and may be shipped using the procedures outlined below.

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Environmental samples should be packed prior to shipment by air using the following procedures:

1. Allow sufficient headspace (ullage) in all bottles (except VOA containers with a septum seal) to compensate for any pressure and temperature changes (approximately 10 percent of the volume of the container).

2. Ensure that the lids on all bottles are tight (will not leak).

3. Place bottles in separate and appropriately sized polyethylene bags and seal the bags. If available, the use of Whirl-Pak bags is preferable, if unavailable seal regular bags with tape (plastic electrical tape).

4. Select a sturdy cooler in good repair. Secure and tape the drain plug with fiber or duct tape inside and outside. Line the cooler with a large heavy duty plastic bag.

5. Place cushioning/absorbent material in the bottom of the cooler and then place the containers in the cooler with sufficient space to allow for the addition of cushioning between the containers.

6. Put "blue ice" (or ice that has been "double bagged" in heavy duty polyethylene bags and properly sealed) on top of and/or between the containers. Fill all remaining space between the containers with absorbent material.

7. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

8. Place the Chain-of-Custody Record or the CLP Traffic Report Form (if applicable) into a plastic bag, and tape the bag to the inner side of the cooler lid.

9. Close the cooler and securely tape (preferably with fiber tape) the top of the cooler shut. Chain-of-custody seals should be affixed to the top and sides of the cooler within the securing tape so that the cooler cannot be opened without breaking the seal.

Region 4 U.S. Environmental Protection Agency Science and Ecosystem Support Division Athens, Georgia	
OPERATING PROCEDURE	
Title: **Logbooks**	
Effective Date: May 30, 2013	**Number:** SESDPROC-010-R5
Authors	
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History	Effective Date
SESCPROC-010-R5, *Logbooks*, replaces SESDPROC-010-R4 **General:** Corrected any typographical, grammatical and/or editorial errors. **Title Page:** Changed author from Liza Montalvo to Hunter Johnson. Changed Enforcement and Investigation Branch Chief from Archie Lee to Danny France. Changed Ecological Assessment Branch Chief from Bill Cosgrove to John Deatrick. Changed Field Quality Manager from Liza Montalvo to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history **Section 1.2**: Added the following statement: "Requirements for SESD records, which include field logbooks, are outlined in the SESD Operating Procedure for Control of Records (SESDPROC-002)." **Section 1.4**: Updated references **Section 2.1**: Added "bound" to the first sentence. Replaced the last sentence of the first paragraph with the following language: "All pertinent field activity information will be recorded contemporaneously when observed or collected to prevent a loss of information." Added the second paragraph partially comprised of language from the omitted paragraph from Section 2.2. **Section2.2**: Omitted the first paragraph. Added the following language to the first line (formerly the second paragraph): "The following requirements apply to **all logbooks**:" Added Items 1 and 2 and omitted Item 4. Renumbered items as appropriate. Added "End of Notes" to the second sentence in item 6 (formerly item 5). Also added the last sentence which states: "Field investigators will draw a diagonal line through blank or unused portions of pages/forms	May 30, 2013

that are located prior to the “End of Notes” entry and initial them.” Added “and SESD-generated forms” to Item 7 (formerly item 6).	
SESCPROC-010-R4, *Logbooks*, replaces SESDPROC-010-R3	October 8, 2010
SESCPROC-010-R3, *Logbooks*, replaces SESDPROC-010-R2	November 1, 2007
SESCPROC-010-R2, *Logbooks*, Replaces SESDPROC-010-R1	September 25, 2007
SESDPROC-010-R1, *Logbooks*, Replaces *Field Records (Logbooks)* SESDPROC-010-R0	August 10, 2007
SESDPROC-010-R0, Field Records (Logbooks), Original Issue	February 5, 2007

TABLE OF CONTENTS

1 General Information

1.1 Purpose

This procedure is to be used by field investigators when documenting pertinent and factual information in logbooks related to field investigations involving sampling and measurement procedures and/or other data collection events.

1.2 Scope/Application

This document describes the various types of information that should be included in the field logbooks used to document field investigations conducted by SESD. Requirements for SESD records, which include field logbooks, are outlined in the SESD Operating Procedure for Control of Records (SESDPROC-002). Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

The procedures found within this document were prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Control of Records, SESDPROC-002, Most Recent Version

SESD Field Branches Quality Management Plan, SESDPLAN-001, Most Recent Version

2 Field Records and Documentation Procedures

2.1 General

Dedicated bound logbooks will be used for field data collection including but not limited to sampling, measurements and observations. Logbook entries should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate. All pertinent field activity information will be recorded contemporaneously when observed or collected to prevent a loss of information.

To facilitate accurate and complete documentation of field sampling and measurement activities, SESD-generated forms may be used. In order to be utilized, SESD-generated forms must be bound prior to use and adhere to all requirements outlined in this procedure. In cases where unbound pages/forms are necessary due to project requirements or practicality, approval must be obtained from both the Field Quality Manager and Management. In these cases, the Field Quality Manager and Management will dictate the steps that will be taken to ensure credibility, traceability and defensibility of information collected.

Any deviations from the quality assurance project plan that occur while in the field will be noted in the logbook(s). Logbook entries that may be considered privileged or confidential information will be handled in accordance with the relevant sections of SESD Operating Procedure for Control of Records (SESDPROC-002). The logbooks will be placed in the SESD project file upon transmittal of the final report to the project requestor.

2.2 Field Data Integrity and Accountability

The following requirements apply to **all logbooks**:

1. The project's unique identifier (unique identification number(s)) will be included on each page.
2. Field personnel will date and number each page. Numbering will be conducted by utilizing a format that incorporates both the current page number and the total number of pages (e.g. "page x of y" or "x/y", where "x" is the current page number and "y" is the total number of pages).
3. Observations, data and calculations will be recorded at the time they are made.
4. Unless prohibited by environmental conditions, pens with permanent ink will be used to record all data. When environmental conditions do not make it feasible to use permanent ink, entries should be made using a non-smear lead pencil (e.g., 2H or 3H). Upon returning from the field, the project leader will photocopy the penciled section of the logbook and certify, in writing, that the photocopied record is a true copy of the original logbook entry. The photocopy will be included in the project file.

5. Entries will be legible and contain pertinent, accurate and inclusive documentation of project activities.
6. Upon completion of the field investigation, the end of project entries in the logbook and/or bound forms will be clearly indicated. This may be accomplished by noting “End” or “End of Notes” on the last page of notes and dating and initialing the notation. Field investigators will draw a diagonal line through blank or unused portions of pages/forms that are located prior to the “End of Notes” entry and initial them.
7. In order to demonstrate continuity of the project and to preclude questioning of the integrity of the data collection process, pages and SESD-generated forms should not be removed from bound logbooks under any circumstances.
8. Data or other information that has been entered incorrectly will be corrected by drawing a line through the incorrect entry and **initialing and dating** the lined-through entry. Under no circumstances should the incorrect material be erased, made illegible or obscured so that it cannot be read.
9. If pre-printed adhesive labels are used in logbooks or bound forms to facilitate organization of information entry, the field investigator who is responsible for taking notes will sign the label with the signature beginning on the label and ending on the page of the logbook such that the label cannot be removed without detection.

2.3 Logbook Entry Information

2.3.1 General Information Required in All Logbooks

The following information will be included either on the front cover or the first page of **all logbooks**:

1. Project name
2. Project location
3. Project identification number
4. Project leader (full name)
5. Sample team leader (full name) and initials
6. Sample team member(s) (full name) and initials

2.3.2 Information Required for Sample Collection

In addition to the information listed in Section 2.3.1, the following information will be included in all logbooks when **samples** are collected:

1. Applicable SESD Operating Procedures for field sampling
2. Date and time of collection
3. Station identification
4. Sample identification

5. Method of collection
6. Number and type of containers
7. Sample collection equipment
8. SESD equipment identification number, if applicable
9. Physical description of sample
10. Matrix sampled
11. Sample team member duties (calibration, collection, deployment, etc.)
12. Sample preservation (including ice), if applicable
13. Conditions that may adversely impact quality of samples, if applicable (rain, wind, smoke, dust, extreme temperature, etc.)
14. GPS coordinates (Non-logging GPS units), if applicable
15. Location of electronic data file backups, if applicable

16. Monitoring of condition of ice in coolers or sampler
17. Other pertinent information.

2.3.3 Information Required for Field Measurements

In addition to the information listed in Section 2.3.1, the following information will be included in all logbooks when **measurements** are conducted:

1. Applicable SESD Operating Procedures for field measurement
2. Date and time of measurement or deployment
3. Sample identification, if appropriate
4. Station identification
5. Sample measurement equipment
6. SESD sample measurement equipment identification number
7. Manufacturer name, lot number and expiration date of all buffers and standards*
8. Calibration information, including before and after calibration readings*
9. Meter end check information
10. Deployment depth and total depth, if applicable
11. Pinger identification number and frequency for deployed equipment, if applicable
12. Time of retrieval for deployed equipment, if applicable
13. Physical description of matrix
14. Sample team member duties (calibration, collection, deployment, etc.)
15. Measurement values for non-logging equipment
16. GPS coordinates (non-logging GPS units), if applicable
17. Location of electronic data file backups, if applicable
18. Ambient air temperature, where applicable
19. Conditions that may adversely impact quality of measurement (Ex. temperature extremes)
20. Maintenance performed, if applicable

21. Meter malfunctions, if applicable
22. Other pertinent information

* Entry of calibration information in logbooks is only required for calibrations conducted in the field. All calibrations conducted at the Field Equipment Center or SESD laboratory will be recorded in the appropriate equipment tracking logbook.

2.3.4 Additional Information for Inclusion

The following information may be included in logbooks as appropriate:

1. Maps/sketches
2. Photographic or videographic log
3. Process diagrams

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Region 4 **U.S. Environmental Protection Agency** **Science and Ecosystem Support Division** **Athens, Georgia**
OPERATING PROCEDURE
Title: **Field Equipment Cleaning and Decontamination**

Effective Date: December 18, 2015	**Number:** SESDPROC-205-R3

Authors
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Approvals
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The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-205-R3, *Field Equipment Cleaning and Decontamination*, replaces SESDPROC-205-R2. **Cover Page:** The author was changed to Brian Striggow. SESD's reorganization was reflected in the authorization section by making John Deatrick the Chief of the Field Services Branch. The FQM was changed from Bobby Lewis to Hunter Johnson. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **General:** Corrected any typographical, grammatical and/or editorial errors. **Section 1.4:** Differentiate between Liquinox® and Luminox® detergents. **Section 3.4:** Restore solvent rinse as alternative cleaning method. **Section 3.7:** Added section on cleaning of 12 Volt electric submersible pumps. **Section 3.8:** Added section on cleaning of bladder pumps. **Section 3.9:** Added language on cleaning and transport of SP15/16 screens **Section 3.10:** Added section on cleaning of rental pumps	December 18, 2015
SESDPROC-205-R2, *Field Equipment Cleaning and Decontamination*, replaces SESDPROC-205-R1.	December 20, 2011
SESDPROC-205-R1, *Field Equipment Cleaning and Decontamination*, replaces SESDPROC-205-R0.	November 1, 2007
SESDPROC-205-R0, *Field Equipment Cleaning and Decontamination*, Original Issue	February 05, 2007

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1 General Information

1.1 Purpose

This document describes general and specific procedures, methods and considerations to be used and observed when cleaning and decontaminating sampling equipment during the course of field investigations.

1.2 Scope/Application

The procedures contained in this document are to be followed when field cleaning sampling equipment, for both re-use in the field, as well as used equipment being returned to the Field Equipment Center (FEC). On the occasion that SESD field investigators determine that any of the procedures described in this section are either inappropriate, inadequate or impractical and that other procedures must be used to clean or decontaminate sampling equipment at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products in this operating procedure does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

- Decontamination: The process of cleaning dirty sampling equipment to the degree to which it can be re-used, with appropriate QA/QC, in the field.

- Deionized water: Tap water that has been treated by passing through a standard deionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard inductively coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. Deionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for deionized water.

- Detergent shall be a standard brand of phosphate-free laboratory detergent such as Liquinox® or Luminox®. Liquinox® is a traditional anionic laboratory detergent and is used for general cleaning and where there is concern for the stability of the cleaned items in harsher cleaners. Luminox® is a specialized detergent with the capability of removing oils and organic contamination. It is used in lieu of a solvent rinse step in cleaning of equipment for trace contaminant sampling. Where not specified in these procedures, either detergent is acceptable.

- Drilling Equipment: All power equipment used to collect surface and sub-surface soil samples or install wells. For purposes of this procedure, direct push is also included in this definition.

- Field Cleaning: The process of cleaning dirty sampling equipment such that it can be returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.

- Organic-free water: Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.

- Tap water: Water from any potable water supply. Deionized water or organic-free water may be substituted for tap water.

1.5 References

SESD Operating Procedure for Management of Investigation Derived Waste, SESDPROC-202, Most Recent Version

SESD Operating Procedure for Equipment Cleaning and Decontamination at the FEC, SESDPROC-206, Most Recent Version

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.6 General Precautions

1.6.1 Safety

Proper safety precautions must be observed when field cleaning or decontaminating dirty sampling equipment. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASPs) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate. At a minimum, the following precautions should be taken in the field during these cleaning operations:

- When conducting field cleaning or decontamination using laboratory detergent, safety glasses with splash shields or goggles, and latex gloves will be worn.

- No eating, smoking, drinking, chewing, or any hand to mouth contact should be permitted during cleaning operations.

1.6.2 Procedural Precaution

Prior to mobilization to a site, the expected types of contamination should be evaluated to determine if the field cleaning and decontamination activities will generate rinsates and other waste waters that might be considered RCRA hazardous waste or may require special handling.

2 Introduction to Field Equipment Cleaning and Decontamination

2.1 General

The procedures outlined in this document are intended for use by field investigators for cleaning and decontaminating sampling and other equipment in the field. These procedures should be followed in order that equipment is returned to the FEC in a condition that will minimize the risk of transfer of contaminants from a site.

Sampling and field equipment cleaned in accordance with these procedures must meet the minimum requirements for the Data Quality Objectives (DQOs) of the study or investigation. If deviations from these procedures need to be made during the course of the field investigation, they will be documented in the field logbook along with a description of the circumstances requiring the use of the variant procedure.

Cleaning procedures for use at the Field Equipment Center (FEC) are found in SESD Operating Procedure for Equipment Cleaning and Decontamination at the FEC (SESDPROC-206).

2.2 Handling Practices and Containers for Cleaning Solutions

Improperly handled cleaning solutions may easily become contaminated. Storage and application containers must be constructed of the proper materials to ensure their integrity. Following are acceptable materials used for containing the specified cleaning solutions:

- Detergent must be kept in clean plastic, metal, or glass containers until used. It should be poured directly from the container during use.

- Tap water may be kept in tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose.

- Deionized water must be stored in clean, glass or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.

- Organic-free water must be stored in clean glass or Teflon® containers prior to use. It may be applied using Teflon® squeeze bottles, or with the portable system.

2.3 Disposal of Cleaning Solutions

Procedures for the safe handling and disposition of investigation derived waste (IDW); including used wash water and rinse water are in SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202).

2.4 Sample Collection Equipment Contaminated with Concentrated Materials

Equipment used to collect samples of concentrated materials from investigation sites must be field cleaned before returning from the study. At a minimum, this should consist of washing with detergent and rinsing with tap water. When the above procedure cannot be followed, the following options are acceptable:

1. Leave with facility for proper disposal;

2. If possible, containerize, seal, and secure the equipment and leave on-site for later disposal;

3. Containerize, bag or seal the equipment so that no odor is detected and return to the SESD.

It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.5 Sample Collection Equipment Contaminated with Environmental Media

Equipment used to collect samples of environmental media from investigation sites should be field cleaned before returning from the study. Based on the condition of the sampling equipment, one or more of the following options must be used for field cleaning:

1. Wipe the equipment clean;

2. Water-rinse the equipment;

3. Wash the equipment in detergent and water followed by a tap water rinse.

4. For grossly contaminated equipment, the procedures set forth in Section 2.4 must be followed.

Under extenuating circumstances such as facility limitations, regulatory limitations, or during residential sampling investigations where field cleaning operations are not feasible, equipment can be containerized, bagged or sealed so that no odor is detected and returned to the FEC without being field cleaned. If possible, FEC personnel should be notified that equipment will be returned without being field cleaned. It is the project leader's responsibility to evaluate the nature of the sampled material and determine the most appropriate cleaning procedures for the equipment used to sample that material.

2.6 Handling of Decontaminated Equipment

After decontamination, equipment should be handled only by personnel wearing clean gloves to prevent re-contamination. In addition, the equipment should be moved away (preferably upwind) from the decontamination area to prevent re-contamination. If the equipment is not to be immediately re-used it should be covered with plastic sheeting or wrapped in aluminum foil to prevent re-contamination. The area where the equipment is kept prior to re-use must be free of contaminants.

3 Field Equipment Decontamination Procedures

3.1 General

Sufficient equipment should be transported to the field so that an entire study can be conducted without the need for decontamination. When equipment must be decontaminated in the field, the following procedures are to be utilized.

3.2 Specifications for Decontamination Pads

Decontamination pads constructed for field cleaning of sampling and drilling equipment should meet the following minimum specifications:

- The pad should be constructed in an area known or believed to be free of surface contamination.
- The pad should not leak.
- If possible, the pad should be constructed on a level, paved surface and should facilitate the removal of wastewater. This may be accomplished by either constructing the pad with one corner lower than the rest, or by creating a sump or pit in one corner or along one side. Any sump or pit should also be lined.
- Sawhorses or racks constructed to hold equipment while being cleaned should be high enough above ground to prevent equipment from being splashed.
- Water should be removed from the decontamination pad frequently.
- A temporary pad should be lined with a water impermeable material with no seams within the pad. This material should be either easily replaced (disposable) or repairable.

At the completion of site activities, the decontamination pad should be deactivated. The pit or sump should be backfilled with the appropriate material designated by the site project leader, but only after all waste/rinse water has been pumped into containers for disposal. See SESD Operating Procedure for Management of Investigation Derived Waste (SESDPROC-202) for proper handling and disposal of these materials. If the decontamination pad has leaked excessively, soil sampling may be required.

3.3 "Classical Parameter" Sampling Equipment

"Classical Parameters" are analyses such as oxygen demand, nutrients, certain inorganic compounds, sulfide, flow measurements, etc. For routine operations involving classical parameter analyses, water quality sampling equipment such as Kemmerers, buckets, dissolved oxygen dunkers, dredges, etc., may be cleaned with the sample water or tap water between sampling locations as appropriate.

Flow measuring equipment such as weirs, staff gages, velocity meters, and other stream gauging equipment may be cleaned with tap water between measuring locations, if necessary.

Note: The procedures described in Section 3.3 are not to be used for cleaning field equipment to be used for the collection of samples undergoing trace organic or inorganic constituent analyses.

3.4 Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds

For samples undergoing trace organic or inorganic constituent analyses, the following procedures are to be used for all sampling equipment or components of equipment that come in contact with the sample:

3.4.1 Standard SESD Method

1. An optional Liquinox® detergent wash step may be useful to remove gross dirt and soil.

2. Clean with tap water and Luminox® detergent using a brush, if necessary, to remove particulate matter and surface films.

3. Rinse thoroughly with tap water.
4. Rinse thoroughly with organic-free water and place on a clean foil-wrapped surface to air-dry.
5. Wrap the dry equipment with aluminum foil or bag in clean plastic. If the equipment is to be stored overnight before it is wrapped in foil, it should be covered and secured with clean, unused plastic sheeting.

3.4.2 Alternative Solvent Rinse Method

The historical solvent rinse method of cleaning equipment for trace contaminant sampling remains an acceptable method.

1. Clean with tap water and Liquinox® detergent using a brush, if necessary, to remove particulate matter and surface films. Equipment may be steam cleaned (Liquinox® detergent and high pressure hot water) as an alternative to

brushing. Sampling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. PVC or plastic items should not be steam cleaned.

2. . Rinse thoroughly with tap water.

3. Rinse thoroughly with deionized water.

4. Rinse with an appropriate solvent (generally isopropanol).

5. Rinse with organic-free water and place on a clean foil-wrapped surface to **a**ir-dry.

4. Wrap the dry equipment with aluminum foil. If the equipment is to be stored overnight before it is wrapped in foil, it should be covered and secured with clean, unused.

3.5 Well Sounders or Tapes

The following procedures are recommended for decontaminating well sounders (water level indicators) and tapes. Unless conditions warrant, it is only necessary to decontaminate the wetted portion of the sounder or tape.

1. Wash with Liquinox® detergent and tap water.

2. Rinse with tap water.

3. Rinse with deionized water.

3.6 Redi-Flo2® Pump

CAUTION – Do not wet the controller. Always disconnect power from the pump when handling the pump body.

The Redi-Flo2® pump and any associated connected hardware (e.g., check valve) should be decontaminated between each monitoring well. The following procedures are required, depending on whether the pump is used solely for purging or used for purging and sampling.

3.6.1 Purge Only (Pump and Wetted Portion of Tubing or Hose)

1. Disconnect power and wash exterior of pump and wetted portion of the power lead and tubing or hose with Liquinox® detergent and water solution.

2. Rinse with tap water.

3. Final rinse with deionized water.

4. Place pump and reel in a clean plastic bag and keep tubing or hose contained in clean plastic or galvanized tub between uses.

3.6.2 Purge And Sample

Grundfos Redi-Flo2® pumps are extensively decontaminated and tested at the FEC to prevent contamination from being transmitted between sites. The relevant sections of SESDPROC-206, *Field Equipment Cleaning and Decontamination at the FEC,* should be implemented in the field where a high risk of cross-contamination exists, such as where NAPL or high-concentration contaminants occur. In most cases, the abbreviated cleaning procedure described below will suffice, provided that sampling proceeds from least to most contaminated areas.

1. Disconnect and discard the previously used sample tubing from the pump. Remove the check valve and tubing adapters and clean separately (See Section 3.6.3 for check valve). Wash the pump exterior with detergent and water.

2. Prepare and fill three containers with decontamination solutions, consisting of Container #1, a tap water/detergent washing solution. Luminox® is commonly used. An additional pre-wash container of Liquinox® may be used; Container #2, a tap water rinsing solution; and Container #3, a deionized or organic-free water final rinsing solution. Choice of detergent and final rinsing solution for all steps in this procedure is dependent upon project objectives (analytes and compounds of interest). The containers should be large enough to hold the pump and one to two liters of solution. An array of 2' long 2" PVC pipes with bottom caps is a common arrangement. The solutions should be changed at least daily.

3. Place the pump in Container #1. Turn the pump on and circulate the detergent and water solution through the pump and then turn the pump off.

4. Place the pump in Container #2. Turn the pump on and circulate the tap water through the pump and then turn the pump off.

5. Place the pump in Container #3. Turn the pump on and circulate deionized or organic-free water through the pump and then turn the pump off.

6. Disconnect power and remove pump from Container #3. Rinse exterior and interior of pump with fresh deionized or organic-free water.

7. Decontaminate the power lead by washing with detergent and water, followed by tap water and deionized water rinses. This step may be performed before washing the pump if desired.

8. Reassemble check valve and tubing adapters to pump. ALWAYS use Teflon® tape to prevent galling of threads. Firm hand-tightening of fittings or light wrench torque is generally adequate.

9. Place the pump and reel in a clean plastic bag.

3.6.3 Redi-Flo2® Ball Check Valve

1. Remove the ball check valve from the pump head. Check for wear and/or corrosion, and replace as needed. During decontamination check for free-flow in forward direction and blocking of flow in reverse direction.

2. Using a brush, scrub all components with detergent and tap water.

3. Rinse with deionized water.

4. Rethread the ball check valve to the Redi-Flo2® pump head.

3.7 Mega-Monsoon® and GeoSub® Electric Submersible Pump

As these pumps have lower velocities in the turbine section and are easier to disassemble in the field than Grundfos pumps, the outer pump housing should be removed to expose the impeller for cleaning prior to use and between each use when used as a sampling pump for trace contaminant sampling.

1. Remove check valves and adapter fittings and clean separately.
2. Remove the outer motor housing by holding the top of the pump head and unscrewing the outer housing from its O-ring sealed seat.
3. Clean all pump components per the provisions of section 3.4. Use a small bottle brush for the pump head passages
4. Wet the O-ring(s) on the pump head with organic-free water. Reassemble the outer pump housing to the pump head.
5. Clean cable and reel per Section 3.4.
6. Conduct final rinse of pump with organic-free water over pump and through pump turbine.

3.8 Bladder Pumps

Bladder pumps are presumed to be intended for use as purge-and-sample pumps. The Geotech® bladder pump and Geoprobe Systems® mechanical bladder pump can be cleaned similarly.

1. Discard any tubing returned with the pump.

2. Completely disassemble the pump, being careful to note the initial position of and retain any springs and loose ball checks.

3. Discard pump bladder.

4. Clean all parts as per the standard cleaning procedure in Section 3.4.

5. Install a new Teflon® bladder and reassemble pump.

3.9 Downhole Drilling Equipment

These procedures are to be used for drilling activities involving the collection of soil samples for trace organic and inorganic constituent analyses and for the construction of monitoring wells to be used for the collection of groundwater samples for trace organic and inorganic constituent analyses.

3.9.1 Introduction

Cleaning and decontamination of all equipment should occur at a designated area (decontamination pad) on the site. The decontamination pad should meet the specifications of Section 3.2 of this procedure.

Tap water brought on the site for drilling and cleaning purposes should be contained in a pre-cleaned tank.

A steam cleaner and/or high pressure hot water washer capable of generating a pressure of at least 2500 PSI and producing hot water and/or steam, with a detergent compartment, should be obtained.

3.9.2 Preliminary Cleaning and Inspection

Drilling equipment should be clean of any contaminants that may have been transported from off-site to minimize the potential for cross-contamination. The drilling equipment should not serve as a source of contaminants. Associated drilling and decontamination equipment, well construction materials, and equipment handling procedures should meet these minimum specified criteria:

- All downhole augering, drilling, and sampling equipment should be sandblasted before use if painted, and/or there is a buildup of rust, hard or caked matter, etc., that cannot be removed by steam cleaning (detergent and high pressure hot water), or wire brushing. Sandblasting should be performed prior to arrival on site, or well away from the decontamination pad and areas to be sampled.

- Any portion of the drilling equipment that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent and high pressure hot water) and wire brushed (as needed) to remove all rust, soil, and other material which may have come from other sites before being brought on site.

- Printing and/or writing on well casing, tremie tubing, etc., should be removed before use. Emery cloth or sand paper can be used to remove the printing and/or writing. Most well material suppliers can provide materials without the printing and/or writing if specified when ordered. Items that cannot be cleaned are not acceptable and should be discarded.

- Equipment associated with the drilling and sampling activities should be inspected to insure that all oils, greases, hydraulic fluids, etc., have been removed, and all seals and gaskets are intact with no fluid leaks.

3.9.3 Drill Rig Field Cleaning Procedure

Any portion of the drill rig, backhoe, etc., that is over the borehole (kelly bar or mast, backhoe buckets, drilling platform, hoist or chain pulldowns, spindles, cathead, etc.) should be steam cleaned (detergent and high pressure hot water) between boreholes.

3.9.4 Field Decontamination Procedure for Drilling Equipment

The following is the standard procedure for field cleaning augers, drill stems, rods, tools, and associated equipment. This procedure does not apply to well casings, well screens, or split-spoon samplers used to obtain samples for chemical analyses, which should be decontaminated as outlined in Section 3.4 of this procedure.

1. Wash with tap water and detergent, using a brush if necessary, to remove particulate matter and surface films. Steam cleaning (high pressure hot water with detergent) may be necessary to remove matter that is difficult to remove with the brush. Drilling equipment that is steam cleaned should be placed on racks or saw horses at least two feet above the floor of the decontamination pad. Hollow-stem augers, drill

rods, etc., that are hollow or have holes that transmit water or drilling fluids, should be cleaned on the inside with vigorous brushing.

2. Rinse thoroughly with tap water.

3. Remove from the decontamination pad and cover with clean, unused plastic. If stored overnight, the plastic should be secured to ensure that it stays in place.

3.9.5 Field Decontamination Procedure for Direct Push Technology (DPT) Equipment

1. Certain specific procedures for the decontamination of DPT tools are described in the various sampling procedures, but the following general guidelines apply:

2. Prior to return to the Field Equipment Center, all threaded tool joints should be broken apart and the equipment cleaned per the provisions of *Section 2.5, Sample Collection Equipment Contaminated with Environmental Media* of this procedure.

3. Equipment that contacts the sample media and is cleaned in the field for reuse should be cleaned per the provisions of *Section 3.4, Sampling Equipment used for the Collection of Trace Organic and Inorganic Compounds* of this procedure. This would include piston sampler points and shoes, screen point sampler screens and sheaths, and the drive rods when used for groundwater sampling.

4. Equipment that does not directly contact the sample media and is cleaned in the field for reuse can generally be cleaned per the provisions of Section *3.7.4, Field Decontamination Procedure for Drilling Equipment* of this procedure.

5. Stainless steel SP15/16 well screens require special care as the narrow slots are difficult to clean under even controlled circumstances and galvanic corrosion can release chrome from the screen surface. As soon as possible after retrieval, the screen slots should be sprayed from the outside to break loose as much material as possible before it can dry in place. To prevent galvanic corrosion, the screens must be segregated from the sampler sheaths, drive rods, and other carbon steel during return transport from the field.

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3.10 Rental Pumps

Completing a groundwater sampling project may require the use of rental pumps. Rental pumps are acceptable where they are of suitable stainless steel and Teflon® construction. These pumps should be cleaned prior to use using the procedures specified herein and a rinse-blank collected prior to use.

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Field Sampling Quality Control**

Effective Date: April 26, 2017	**Number:** SESDPROC-011-R5

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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-011-R5, *Field Sampling Quality Control*, replaces SESDPROC-011-R4 **Cover Page:** SESD's reorganization was reflected in the authorization section by making John Deatrick the Chief of the Field Services Branch. The FQM was changed from Bobby Lewis to Hunter Johnson. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history. **General:** Corrected any typographical, grammatical and/or editorial errors. Changed name of Enforcement and Investigations Branch to Field Services Branch. Removed references to Ecological Assessment Branch. Added Section 2.9 to the Table of Contents. **Section 1.4.6:** Added definition for Organic-Free Water **Section 3.5:** Changed volume needed for soil MS/MSD samples from triple to double volume. **Section 4.1:** Modified statement to read**:** Each lot of chemical preservative will be tested for the appropriate analytes by either FEC Staff or the Branch QAO.	April 26, 2017
SESDPROC-011-R4, *Field Sampling Quality Control*, replaces SESDPROC-011-R3	February 5, 2013
SESDPROC-011-R3, *Field Sampling Quality Control*, replaces SESDPROC-011-R2	October 15, 2010
SESDPROC-011-R2, *Field Sampling Quality Control*, replaces SESDPROC-011-R1	January 28, 2008
SESDPROC-011-R1, *Field Sampling Quality Control*, replaces SESDPROC-011-R0.	October 19, 2007
SESDPROC-011-R0, *Field Sampling Quality Control*, Original Issue	February 5, 2007

TABLE OF CONTENTS

1 General Information

1.1 Purpose

This document describes procedures established to ensure the quality of SESD field sampling activities, including Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations. Collectively, these procedures ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures, resulting in the collection of representative samples. Strict adherence to these procedures forms the basis for an acceptable field sampling quality assurance program.

1.2 Scope/Application

The procedures contained in this document are to be used by field investigators when collecting and handling samples in the field and when preparing sampling equipment for SESD field investigations. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD local area network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 Definitions

1.4.1 Sample

A part of a larger lot, usually a volume, area, period or population.

1.4.2 Variability

The range or "distribution" of results around the mean value obtained from samples within a population. There are three types of variability which should be measured or otherwise accounted for in field sampling, depending on the data quality objectives (DQO) for the study:

1. Temporal Variability

   Temporal variability is the range of results due to changes in contaminant concentrations over time. An example would be the range of concentrations obtained for a given parameter in wastewater samples collected at different times from an outfall where contaminant concentrations vary over time.

2. Spatial Variability

   Spatial variability is the range of results due to changes in contaminant concentrations as a function of their location. An example would be the range of concentrations obtained for a given parameter in surface soil from a site where discreet "hot spots" are present due to localized releases of contaminants on otherwise uncontaminated soil.

3. Sample Handling Variability

   Sample handling variability is the range of results due to the sample collection and handling techniques used by the sampler. This variability manifests itself as a positive bias due to errors such as unclean sampling equipment, cross contamination, etc., or a negative bias due to improper containers or sample preservation.

1.4.3 Grab Sample

An individual sample collected from a single location at a specific time or period of time. Grab samples are generally authoritative in nature.

1.4.4 Composite Sample

A sample collected over a temporal or spatial range that typically consists of a series of discrete, equal samples (or "aliquots") which are combined or "composited." A composite sample represents the average characteristics of the population under consideration. Four types of composite samples are listed below:

1. Time Composite (TC) – a sample comprised of a varying number of discrete samples or "aliquots" collected at equal time intervals during the compositing period. The TC sample is typically used to sample wastewater or streams.

2. Flow Proportional Composite (FPC) – A sample consisting of discrete samples or "aliquots" collected at a rate proportional to flow. The aliquots are collected during the compositing period by either a time-varying/constant volume (TV/CV) method ("automated flow proportioning") or a time-constant/varying volume (TC/VV) method ("manual flow proportioning"). The TV/CV method is typically used with automatic samplers that are paced by a flow meter. The TC/VV method is a manual method that individually proportions a series of discretely collected aliquots. The FPC is

typically used when sampling wastewater.

3. Areal Composite – a sample composited from individual, equal aliquots collected on an areal or horizontal cross-sectional basis. Each aliquot is collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.

4. Vertical Composite – a sample composited from individual, equal aliquots collected from a vertical cross section. Each aliquot is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries.

1.4.5 De-ionized Water

Tap water that has been treated by passing it through a standard de-ionizing resin column. At a minimum, the finished water should contain no detectable heavy metals or other inorganic compounds (i.e., at or above analytical detection limits) as defined by a standard Inductively Coupled Argon Plasma Spectrophotometer (ICP) (or equivalent) scan. De-ionized water obtained by other methods is acceptable, as long as it meets the above analytical criteria. Organic-free water may be substituted for de-ionized water.

1.4.6 Organic-Free Water

Tap water that has been treated with activated carbon and deionizing units. At a minimum, the finished water must meet the analytical criteria of deionized water and it should contain no detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined by the Region 4 laboratory for a given set of analyses. Organic-free water obtained by other methods is acceptable, as long as it meets the above analytical criteria.

1.4.7 Branch Field Equipment Manager

Staff, designated by management, who are responsible for ensuring that the procedures for Equipment Inventory and Management are followed. At least one Branch Field Equipment Manager will be designated for the Field Services Branch (FSB).

1.5 References

SESD Safety, Health and Environmental Management Program (SHEMP) Manual, Most Recent Version

SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005-most recent version)

SESD Operating Procedure for Competency and Proficiency Testing, (SESDPROC-006, most recent version)

SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108-most recent version)

SESD Operating Procedure for Sediment Sampling (SESDPROC-200-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205-most recent version)

SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206-most recent version)

SESD Operating Procedure for Soil Sampling (SESDPROC-300-most recent version)

SESD Operating Procedure for Waste Sampling (SESDPROC-302-most recent version)

USEPA Region 4 Environmental Investigations Standard Operating Procedures and Quality Assurance Manual (EISOPQAM), November 2001

USEPA Region 4 Analytical Support Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM), Most Recent Version

Loan-In Form (SESDFORM-011, most recent version)

SESD Operating Procedure for Packing, Marking, Labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209, most recent version)

SESD Operating Procedure for Training (SESDPROC-007-most recent version)

SESD Operating Procedure for Corrective Action (SESDPROC-009-most recent version)

SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101-most recent version)

2 Field Sampling Quality Control Considerations

This section provides guidelines for establishing quality control procedures for sampling activities. Strict adherence to all of the standard operating procedures outlined in this subsection forms the basis for an acceptable sampling quality assurance program.

2.1 Experience Requirements

There is no substitute for field experience. This field experience will be gained by on-the-job training using the "buddy" system. Each new investigator will accompany an experienced employee on as many different types of field studies as possible. During this training period, the new employee will be permitted to perform all facets of field investigations, including sampling, under the direction and supervision of senior investigators. Specific requirements covering experience, competency and proficiency are found in the SESD Operating Procedure for Competency and Proficiency Testing (SESDPROC-006) and SESD Operating Procedure for Training (SESDPROC-007).

2.2 Traceability Requirements

All sample collection and measurement activities will be traceable through field records to the person collecting the sample or making the measurement. All maintenance and calibration records for sampling and measurement equipment (where appropriate) will be kept so that they are similarly traceable. The SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108) contain specific procedures to be followed that ensure traceability.

2.3 Chain-of-Custody

Specific chain-of-custody procedures are included in SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005). These procedures will ensure that evidence collected during an investigation will withstand scrutiny during litigation. To assure that procedures are being followed, it is recommended that field investigators or their designees audit chain-of-custody entries, tags or labels, field notes, and any other recorded information for accuracy. Additionally, the SESD FQM will randomly conduct reviews of project files to ensure that quality procedures are being followed.

2.4 Sampling Equipment Construction Material

Sampling equipment construction materials can affect sample analytical results. Field investigators will ensure the sample equipment construction material will not introduce contaminants to the sample being collected.

2.5 Sample Preservation

Samples for some analyses must be preserved in order to maintain their integrity. Preservatives required for routine analyses of samples collected are found in the USEPA Region 4 Analytical Services Branch Laboratory Operations and Quality Assurance Manual (ASBLOQAM). Chemical preservatives used will be supplied by the Region 4 laboratory or purchased by the Branch Field Equipment Manager. All samples requiring preservation should be preserved immediately upon collection in the field. Records of sample preservation, including ice, will be documented in the field log books.

Samples that **should not** be preserved in the field are:

1. Those collected within a hazardous waste site that are known or thought to be highly contaminated with toxic materials which may be highly reactive. Barrel, drum, closed container, spillage or other source samples from hazardous waste sites are not to be preserved with any chemical.

2. Those that have extremely low or high pH or samples that may generate potentially dangerous gases if they were preserved according to the ASBLOQAM.

All samples preserved with chemicals will be clearly identified by indication on the sample tag or label that the sample is preserved. If samples normally requiring preservation were not preserved, field records should clearly specify the reason. Samples shipped by air will not be preserved with nitric acid, hydrochloric acid, sodium hydroxide or sulfuric acid in excess of the amount specified in the ASBLOQAM.

2.6 Sample Collection Precautions

In order to prevent cross-contamination during sample collection, the following precautions will be taken:

1. A clean pair of new, non-powdered, disposable latex or nitrile gloves will be worn each time a different location is sampled and the gloves should be donned immediately prior to sampling. The gloves should not come into contact with the media being sampled.

2. Sample containers for source samples or samples suspected of containing high concentrations of contaminants will be placed in separate plastic bags immediately after collecting, tagging, etc.

3. If possible, environmental (low concentration) samples and source or waste samples (high concentration) should be collected by different field teams. If different field teams cannot be used, all environmental samples should be collected first and placed in separate ice chests or shipping containers. Samples of waste or

highly contaminated samples should never be placed in the same ice chest as environmental samples. Ice chests or shipping containers for source or waste samples or any samples suspected to contain high concentrations of contaminants will be lined with new, clean, plastic bags.

4. If possible, one member of the field sampling team should record all of the field notes, collect GPS data, etc., while the other members collect the samples.

5. When sampling surface water and sediment at the same location, the water sample should always be collected before the sediment sample is collected.

6. Sample collection activities should proceed progressively from the least suspected contaminated area to the most suspected contaminated area.

7. Investigators should use equipment constructed of Teflon®, stainless steel, or glass that has been properly pre-cleaned according to either the SESD Operating Procedure for Field Equipment Cleaning and Decontamination (SESDPROC-205) or the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) for collection of samples for trace metals or organic compounds analyses. Teflon® or glass is preferred for collecting samples where trace metals are of concern. Equipment constructed of plastic or PVC should <u>not</u> be used to collect samples for trace organic compounds analyses.

8. Field investigators should ensure the sample containers they are using have been verified as suitable for the analyses that will be conducted on the samples through the quality control procedures discussed in Section 4 of this procedure.

Upon returning from the field, un-used sample containers will be examined by project leaders to determine whether bottles should be discarded, recycled or re-shelved for use on other projects. A load-in form (SESDFORM-011) will be completed and signed by project leaders to identify the future use of sample containers returning from the field. Opened boxes of sampling containers that can be re-used, will be segregated from sealed boxes of new containers.

Opened bags of latex or nitrile gloves returning from the field will be segregated from unopened gloves and will not be re-used for sample collection on other projects.

2.7 Sample Handling and Mixing

Once a sample has been collected, it may have to be transferred into separate containers for different analyses. Sample transfer should be done as soon as possible. If necessary, aqueous samples may be collected into a single, larger container for homogenization and transferred into individual sample containers. However, aqueous samples collected for volatile organic compounds, oil and grease, bacteria, sulfides and phenols analyses may not be transferred using this procedure.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is representative of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

1. The material in the sample pan should be divided into quarters and each quarter should be mixed individually.

2. Two quarters should then be mixed to form halves.

3. The two halves should be mixed to form a homogenous matrix.

This procedure should be repeated several times until the sample is adequately mixed. If round bowls are used for sample mixing, adequate mixing is achieved by stirring the material in a circular fashion, reversing direction and occasionally turning the material over.

2.8 Special Handling of Samples for Volatile Organic Compounds Analysis

Water samples to be analyzed for volatile organic compounds should be stored in 40-ml septum vials with screw cap and Teflon®-silicone disk in the cap to prevent contamination of the sample by the cap. The disks should be placed in the caps (Teflon® side to be in contact with the sample) in the laboratory prior to the beginning of the field investigation.

The vials should be completely filled to prevent volatilization, and extreme caution should be exercised when filling a vial to avoid any turbulence which could also produce volatilization. The sample should be carefully poured down the side of the vial to minimize turbulence. As a rule, it is best to gently pour the last few drops into the vial so that surface tension holds the water in a convex meniscus. The cap is then applied and some overflow is lost, but the air space in the bottle is eliminated. After capping, turn the bottle over and tap it to check for bubbles. If a bubble or bubbles are present, the vial should be topped off using a minimal amount of sample to re-establish the meniscus. Care should be taken not to flush any preservative out of the vial during topping off. If, after topping off and capping the vial, bubbles are still present, a new vial should be obtained and the sample re-collected.

Soil and sediment samples for VOC analyses should be collected and handled as specified in the SESD Operating Procedure for Soil Sampling (SESDPROC-300), Waste Sampling (SESDPROC-302) or the SESD Operating Procedure for Sediment Sampling (SESDPROC-200). Soil and sediment samples collected for VOC analyses should <u>not</u> be mixed.

2.9 Sample Storage and Transport

After collection, sample handling should be minimized. Field investigators should use extreme care to ensure that samples are not contaminated during storage. Environmental and waste samples are typically stored in coolers. To reduce the risk of cross contamination, smaller sample containers such as 8 ounce glass jars, 40 ml VOA vials, and one-liter amber bottles should be placed inside of sealed, plastic bags before being placed in the cooler. If ice is required for preservation of the samples, the ice should be contained in a plastic bag or some equivalent container to prevent the potential for cross contamination of the samples by water produced from melting ice. If ice is used, the coolers should be checked regularly and water should be drained as needed. Custody of samples will be maintained according to the SESD Operating Procedure for Sample and Evidence Management (SESDPROC-005).

Samples will either be transported to the analytical laboratory by field investigators or shipped by common carrier. Shipping of samples will be conducted in accordance with the SESD Operating Procedure for Packing, Marking, labeling, and Shipping of Environmental and Waste Samples (SESDPROC-209).

3 Quality Control Samples

Quality control samples are collected during field studies for various purposes, among which are to isolate site effects (control samples), to define background conditions (background sample), and to evaluate field/laboratory variability (spikes and blanks, trip blanks, duplicate, split samples, etc.).

3.1 Control Sample

A control sample is typically a discrete grab sample collected to isolate a source of contamination. Isolation of a source could require the collection of both an upstream sample at a location where the medium being studied is unaffected by the site being studied, as well as a downstream control which could be affected by contaminants contributed from the site under study.

3.2 Background Sample

A background sample (usually a grab sample) is collected from an area, water body or site similar to the one being studied, but located in an area known or thought to be free from pollutants of concern.

3.3 Variability Samples

Variability may be defined as a variation in concentrations of compounds or analytes across a site or area of investigation or variations, across time, of waste streams or surface water bodies. Variation can also be introduced during sample handling. The following procedures are used to assess and evaluate variability. When appropriate, spatial duplicate grab and/or composite samples should be collected during investigations and studies in accordance to the project DQOs. In general, no more than ten percent of all samples should be collected as spatial duplicates.

3.3.1 Spatial Variability Duplicate

The following spatial duplicate sampling procedures should be used during the collection of samples as a measure of variability within the area represented by the sample. These samples should be collected at the same time, using the same procedures, the same type of equipment, and in the same types of containers as the original samples. They should also be preserved in the same manner and submitted for the same analyses as the required samples.

Spatial variability duplicate samples are typically collected during investigations where samples are collected from grids that are positioned at fixed intervals over the study area and a sample collection pattern is established within the grids. Spatial variability duplicate samples are collected using the same compositing pattern as the original sample and are collected within the same general area of representativeness, however the pattern is shifted relative to the original aliquot locations. This amount and direction of shift for the duplicate sample is dependent upon the size of the grid or area being sampled and should be specified in the QAPP for the investigation. Data from spatial duplicates will be examined by the investigation project leader to determine if the observed spatial variability is acceptable, based on the investigation or study objectives.

3.3.2 Temporal Variability Duplicate

When appropriate, temporal variability at a given sampling location will be measured by collecting temporal duplicate samples. These samples will be collected from the same sampling location, using the same techniques and the same type of equipment, but at a time different from the original sample. The time selected for the temporal duplicate sample will be similar to the time or span of time specified for the original sample in the project work plan. Data from temporal duplicates will be examined by the project leader to determine if samples represent the time span intended in the project work plan.

3.3.3 Sample Handling Variability

The effectiveness of sample handling techniques will be measured by collecting split and blank samples.

<u>Split Samples</u>

Split samples will be collected by initially collecting twice as much volume as is normally collected. The material will be apportioned, after mixing, if appropriate, into two sets of containers. Both sets of containers will be submitted for analyses with one set designated as an "original sample," the other designated as a "split sample." Data from the split samples will be examined by the project leader to assess sample handling variability. On large studies (more than 20 samples collected), a minimum of 5 percent, but no more than 10 percent, of all samples will be collected as split samples unless required by site data quality objectives.

<u>Blank Samples</u>

The following blank samples will be prepared by the laboratory and obtained by the project leader prior to traveling to a sample site.

1. Water Sample VOC Trip Blank - A water sample VOC trip blank is required for every study where water samples are collected for VOC analysis. Sealed preserved (or unpreserved, if unpreserved vials were used during the investigation) 40-ml VOC vials will be transported to the field. Two sealed VOC vials will be submitted per trip blank sample. At least one trip blank sample will be submitted per sample shipment. Trip blanks will be prepared by lab personnel. Investigators should submit their request for trip blanks at least one week in advance of scheduled field investigations and inspections and never

(except in emergency situations) less than two days in advance of scheduled field investigations and inspections. These samples should not be picked up earlier than the morning of departure for the scheduled inspection/investigation. These trip blanks will be handled and treated in the same manner as the water samples collected for volatile organic compounds analysis on that particular study. These samples will be clearly identified on sample labels and Chain-of-Custody Records as trip blanks.

2. Soil/Sediment Sample VOC Trip Blank - A soil/sediment sample VOC trip blank is required for every study where soil and/or sediment samples are collected for VOC analysis. The required containers are specified the USEPA Region 4 ASBLOQAM. The request and pick up of the soil blank sample will be the same as for the water trip blank. En Core® containers will be transported to the field. These blanks will be handled and treated by field investigators in the same manner as the soil samples collected for VOC analysis on that particular study. These samples will be clearly identified on sample labels and Chain-Of-Custody Records as trip blanks. Two sealed En Core® containers will be submitted per trip blank sample. At least one set of trip blank samples will be submitted per sample shipment.

The following blanks are prepared in the field:

1. Sample Preservative Blanks - SESD will generally use chemical preservatives stored in individual single-use vials. The chemical preservative will be tested prior to use for the appropriate analytes. The use of pre-tested, single-use vials eliminates the need to routinely collect preservative blanks in the field. If the preservatives are stored in containers that will be used to preserve multiple samples, blanks will be collected to evaluate the potential for cross-contamination resulting from the preservation process. If preservative blanks are collected, sample containers will be filled with de-ionized water by SESD personnel and transported to the field and preserved and submitted for the same analyses as the other inorganic samples collected. These samples will be clearly identified as preservatives blanks on sample labels and the Chain-Of-Custody Record(s). At least one preservative blank for each type of preserved sample should be collected at the end of routine field investigations. In addition, one preservative blank will be collected for each multi-use bottle of preservative used.

   Note: The deionized water will be generated from a water treatment unit provided by the SESD laboratory.

2. Equipment Rinsate Blanks - Equipment rinsate blanks will be collected whenever field decontamination of equipment to be re-used in sampling activities is performed.

   When field cleaning of equipment is required during a sampling investigation, a piece of the field-cleaned equipment will be selected for collection of a rinse blank. At least one rinse blank will be collected during each week of sampling operations. After the piece of equipment has been field cleaned and prior to its being used for sample operations, it will be rinsed with organic-free water. The rinse water will be collected and submitted for analyses of all constituents for which normal samples collected with that piece of equipment are being analyzed.

3. Organic-Free Water System Blanks - When using a portable organic-free water generating system in the field, a sample of the water generated by the system will be collected at least once during each week of operations. Based on the objectives of the study or investigation, it may be appropriate to collect a sample of the raw source water. The collected water sample will be submitted for analyses of all constituents for which normal samples are being analyzed.

4. Material Blanks - When construction materials are being used on a site in such a way as to have a potential impact on constituent concentrations in the sample, a sample of each material will be submitted for analysis.

   Note: For drilling operations where materials are shipped directly to the site from the supplier, see SESD Guidance for Design and Installation of Monitoring Wells (SESDGUID-101) for material blank collection and reporting requirements.

5. Automatic Sampler Blanks - In general, cleaning procedures outlined in the SESD Operating Procedure for Field Equipment Cleaning and Decontamination at the FEC (SESDPROC-206) should be adequate to ensure sample integrity. However, it is the standard practice of the Field Services Branch to submit automatic sampler blanks for analyses when automatic samplers are used to collect samples for organic compounds and metals analyses. Automatic sampler blanks for other standard analyses may be submitted in the event of a special investigation (e.g., criminal or civil).

6. Field Blank - A field blank is a sample that is prepared in the field to evaluate the potential for contamination of a sample by site contaminants from a source not associated with the sample collected (for example air-borne dust or organic vapors which could contaminate a soil sample). Organic-free water is taken to the field in sealed containers or generated on-site. The water is poured into the appropriate sample containers at pre-designated locations at the site. Field blanks should be collected in dusty environments and/or from areas where volatile organic contamination is present in the atmosphere and originating from a source other than the source being sampled.

7. Temperature Blank - A temperature blank is a container of water shipped with each cooler of samples requiring preservation by cooling to 6°C (ice). The temperature of the blank is measured at the time of sample receipt by the laboratory. No temperature blank is necessary for waste samples since waste samples do not require ice for preservation.

8. Wipe Sample Blank - A wipe sample blank is a sample of the material and solvent used for collecting wipe samples. The blank is handled, packaged and transported in the same manner as all other wipe samples with the exception that it is not exposed to actual contact with the sample medium.

9. Water Filter Blank - When filters are used for sampling a dissolved constituent, de-ionized water should be run through at least one filter from each lot and the filtered water submitted for the same analyses. When filters are used for chlorophyll sampling, the filter should be prepared using de-ionized water and submitted for the same chlorophyll analysis.

3.4 Spikes

Spike samples are used to measure bias due to sample handling or analytical procedures. Spike samples are typically used by SESD to evaluate the performance of contract laboratories and are shipped directly to the CLP laboratory by the ESAT contractor.

3.5 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Organic Compounds Analyses

Matrix spike and matrix spike duplicate (MS/MSD) samples will be submitted to the laboratory for volatile organic compounds, extractable organic compounds, pesticides/PCBs and/or herbicides analyses from at least one sampling location per project and laboratory used. One MS/MSD sample should be collected per 20 samples per media collected.

Additional volume will be required for the soil MS/MSD samples. Semi-volatile organic compounds, pesticides, and PCB analyses of soil/sediment samples require the collection of one additional eight ounce glass jar. For VOC soil/sediment samples, double volume, i.e., six En Cores® or six 40 ml vials with syringe collected sample, is needed for the MS/MSD samples.

Additional volume will be required for the water MS/MSD samples. For routine full scan analysis, i.e., extractable organic compounds, pesticides and PCBs, four one-liter amber containers provide the required sample volume. Eight containers, therefore, should be submitted for the MS/MSD sample. For VOC water samples, a total of six 40 ml vials should be collected.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. The duplicate samples should be clearly identified as "Duplicate Sample for Matrix Spike" or “MS/MSD” on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.6 Matrix Spike/Matrix Spike Duplicate Samples for Water and Soil Samples for Inorganic Analyses

A matrix spike sample and a duplicate sample (MS/MSD) will be submitted to the laboratory for inorganic analyses from at least one sampling location per project and laboratory used. One matrix spike and duplicate sample should be collected per 20 samples per media collected per laboratory.

Soil/sediment and water samples collected for inorganic analyses will normally have sufficient sample volume to perform the matrix spike analyses without requiring the collection of extra sample volume. The project leader should designate a sample, typically one considered to be representative of background or relatively uncontaminated conditions, as the matrix spike sample. For water samples, the sample volume collected will normally provide adequate volume for the MS/MSD analyses.

MS/MSD samples should be collected from a location expected to be relatively free from contamination, since the samples will be used for laboratory quality control purposes. MS/MSD samples should be clearly identified as "Duplicate Sample for Matrix Spike" or "MS/MSD" on the Chain-Of-Custody Record, in the field logbook and on the Contract Laboratory Program (CLP) Traffic Report Form (if appropriate). This procedure will be followed for all projects where water samples are collected for the indicated analyses. For non-routine sampling events, the Region 4 SESD laboratory should be consulted for specific sample volume and container requirements.

3.7 Special Quality Control Procedures for EPA Contract Laboratories

On a case-by-case basis, field investigators may be required to collect split samples (or duplicate samples if appropriate) for analyses by either the Region 4 SESD laboratory or contract laboratories. The split samples are to be submitted to the Region 4 laboratory using established procedures. The contract laboratory involved <u>will not be notified</u> that samples were split, i.e., there should be no indication on Chain-Of-Custody Records or CLP Traffic Report Forms submitted to the contract laboratories that these samples were split with the Region 4 SESD laboratory.

3.8 Special Quality Control Procedures for Dioxins and Furans

The Region 4 laboratory does <u>not</u> conduct in-house analyses for dioxins and furans. Dioxin and furans analyses are conducted by contract laboratories. The Region 4 laboratory may accept environmental samples (soil, sediment, groundwater and surface water) suspected of being contaminated with polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF), as long as suspected PCDD and PCDF contamination is not due to RCRA hazardous waste classified as F020-023 and/or F026-

028. If these environmental samples are not contaminated with an F020-023 and/or F026-028 waste, it may be analyzed for parameters other than dioxin and furans. Environmental samples known or suspected to be contaminated with the RCRA hazardous waste F020-023 and or F026-028 will not be accepted.

NOTE: Environmental samples suspected of being contaminated with RCRA hazardous waste classified in 40 CFR, 261.31 as F032 will be accepted. The F032 waste is defined as wastewaters (except those that have not come into contact with process contaminants), process residuals, preservative drippage, and spent formulations from wood preserving processes generated at plants that currently use or have previously used chlorophenolic formulations. The F032 listing does not include K001 bottom sediment sludge from the treatment of wastewater from wood preserving processes that use creosote and or pentachlorophenol. Prior to a sampling event, the project leaders should consult with the Analytical Services Branch Sample Control Coordinator to determine if the Region 4 laboratory can accept the samples. The Region 4 SESD laboratory should also be consulted for the current quality control procedures for dioxins and furans samples prior to a sampling investigation.

4 Internal Quality Control Procedures

The focus of this section is on Field Equipment Center (FEC) operations involving preparation of sampling and support equipment for field operations, as well as, field data generated under the specific sample collection quality control procedures discussed in Section 2. Quality control checks of these operations ensure that field sampling teams are provided with equipment that is suitable for sampling use, and that field sampling is conducted using proper procedures.

4.1 Traceability Requirements

Records, will be kept by designated SESD staff or FEC personnel documenting the dates of operations and the person performing operations for the following:

1. Organic-Free Water System Maintenance (FEC System) - Maintenance on the FEC organic-free water system will be performed at least once per 180 days.

2. Air Monitoring Safety Instrumentation Checkouts - Pre-loadout checks on safety monitoring instrumentation will be recorded each time they are performed. Discrepancies will be immediately reported to the Branch Safety Officer.

3. Self Contained Breathing Apparatus (SCBA) Checkouts - Pre-loadout checks on SCBAs will be recorded when they are performed. SCBA checkouts will be performed at least once per calendar quarter in the absence of loadout requests. Any discrepancies will be reported immediately to the Branch Safety Officer.

4. Other Equipment Maintenance - Maintenance performed on equipment other than that listed above will be accordance to the SESD Operating Procedure for Equipment Inventory and Management (SESDPROC-108). All required repairs will be reported to appropriate Branch Field Equipment Manager.

5. Tubing, Sampling Containers and Latex Gloves - The Field Services Branch Quality Assurance Officer (FSB QAO) is responsible for conducting verification sampling for tubing, sample containers, and latex gloves that are used during field investigations. Upon receipt, the tubing, containers and gloves are placed in the quarantine room at the FEC. A record is kept of the lot numbers for each shipment received. The FSB QAO, or designee, will collect blank samples from tubing, containers and gloves within each lot received and will review the results to ensure the sample containers and gloves are suitable for use during field investigations. Once the supplies are deemed suitable, the FSB QAO will release the items for use.

6. Chemical preservatives commercially purchased will be tested prior to use. Each lot of chemical preservative will be tested for the appropriate analytes by either the

Branch Field Equipment Manager or the FSB QAO. Once released by FSB QAO, the preservatives can be used in the field.

7. Equipment - All equipment cleaned and wrapped for field use will be marked with the date on which preparation was completed. Equipment will be stored at the FEC in specified areas to minimize the risk of contamination while awaiting use.

4.2 Specific Quality Control Checks

When collecting samples during field investigations, it is necessary to take measures to prevent cross contamination to ensure the integrity of the data generated. The field branches conduct verification sampling of sample containers, gloves, sampling equipment, tubing and water utilized during field investigations as one of these measures. At least once per calendar quarter, the FSB QAO will conduct the following checks and issue a written report to the Field Quality Manager with the results.

1. Collect and submit for analyses samples of each new lot of containers received, Bottles from each lot will be tagged and sealed, then submitted for the following analyses:

    1. 1-liter Amber – extractable organics, pesticides, and PCBs.

    2. 8-oz. Clear Glass – metals, cyanide, extractable organics, pesticides, PCBs, and volatile organic compounds.

    3. 1-Liter Polyethylene – metals and cyanide.

    NOTE: In addition to the quality control checks listed above, samples may be collected during field investigations for classical inorganic parameters such as nitrates, nitrites, sulfides, etc. Due to the detection levels generally required for these parameters, it is unlikely that cross contamination may occur in association with the sample containers and sampling equipment used during sample collection. Therefore, classical inorganic analyses are not conducted as part of the routine quality control checks. If the data quality objectives require additional quality control checks, bottles will be submitted to the laboratory for analyses.

2. Collect and submit for analyses a rinsate blank for each new lot of latex or nitrile gloves received during the calendar quarter. Samples will be collected as rinse blanks using organic-free water. The rinsate will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. A new glove will be rinsed for each parameter (e.g., one glove for the VOC sample, another glove for metals, etc.) to avoid dilution of potential contaminants on the gloves. Water for the VOC samples should be provided by the ASB laboratory.

3. Collect and submit for analyses a sample of water from the FEC organic-free water system. The sample will be submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs.

4. Collect and submit for analyses a rinsate blank of at least one piece of sampling or sample related equipment stored at the FEC. The sample will submitted for analyses of VOCs, metals, cyanide, extractable organics, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

5. Collect and submit for analyses a rinsate blank for each new lot of Silastic® or Tygon® tubing used in peristaltic pump head. The sample will be submitted for metals and cyanide analysis.

6. Teflon® tubing – Collect and submit for analyses a rinsate blank for each new lot of Teflon® tubing received. Rinse blanks will be collected through the Teflon tubing. The sample will be submitted for metals, cyanide, extractable organics, volatile organic compounds, pesticides and PCBs. Water for the VOC samples should be provided by the ASB laboratory.

4.3 Quality Control for Special Order Equipment and Supplies

Some equipment and supplies ordered for specific projects are received in what can be considered ready to use condition. In order to ensure the integrity of these materials, an equipment rinsate blank will be collected from at least one item in each lot. The equipment and supplies will not be used until the QAO has reviewed the analytical data for the blanks and released the items.

4.4 Quality Control Evaluation and Corrective Action

All field investigation reports will contain a clearly identified section where the results for all field generated quality control (QC) samples are discussed and reported. Quality control data review includes but is not limited to detections of organic and inorganic compounds at any concentration in quality control blanks (i.e., trip blanks, equipment rinsate blanks, portable organic-free water system blanks, etc.).

All detections of organic and inorganic compounds will be immediately reported to the FSB QAO. The project leader will analyze of the results to determine if the source of contamination can be identified. If the source of contamination cannot be determined by the project leader, the branch QAO will conduct an additional review of the results to assess the source of contamination. If the source of contamination cannot be determined, the QAO will monitor all quality control results generated by the branch and assess the data for trends of contamination.

If it is determined by the project leader and the FSB QAO that the contamination adversely impacts the data collected during the investigation, the project leader will report the results to their Section Chief and the FQM. The project leader, in consultation with management, will determine whether the impacted data are usable or should be rejected. If data are rejected, the project leader and their management will determine whether samples must be recollected.

Data reported to the FQM will be analyzed to determine if the contamination is due to non-conforming work. If it is determined by the FQM, in consultation with management, that the contamination is due to non-conforming work, a corrective action is warranted and will be selected and implemented in a timely manner. If a corrective action is required, it must be implemented and reported according to the SESD Operating Procedure for Corrective Action (SESDPROC-009). If contamination is not due to non-conforming field work, then the source of contamination will be identified, if possible, and documented by the FQM. If the source of contamination cannot be determined, FQM will monitor all quality control results generated by SESD and assess the data for trends of contamination.

4.4.1 Quality Assurance Reports

It is each project leader's responsibility to ensure that a copy of the quality assurance data from each field investigation report is provided to the FSB QAO, who will compile a quarterly report of field quality assurance data and forward the report to the FQM.

The FQM will prepare an annual quality assurance report based on the quarterly reports. This report will be distributed to all field investigators each year and will document and discuss all quality control issues or trends identified during the data review. This report will be retained by the FQM to document that QC measures have been taken, that the QC measures are appropriate, that the QC results are acceptable or, if not, that corrective actions were taken.

COPY

Region 4
U.S. Environmental Protection Agency
Science and Ecosystem Support Division
Athens, Georgia

OPERATING PROCEDURE

Title: **Management of Investigation Derived Waste**	
Effective Date: July 3, 2014	**Number:** SESDPROC-202-R3
Authors	
Name: Art Masters Title: Environmental Scientist, Regional Expert Signature:	Date: 7/2/14
Approvals	
Name: John Deatrick Title: Acting Chief, Enforcement and Investigations Branch Signature:	Date: 7/2/14
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Revision History

The top row of this table shows the most recent changes to this controlled document. For previous revision history information, archived versions of this document are maintained by the SESD Document Control Coordinator on the SESD local area network (LAN).

History	Effective Date
SESDPROC-202-R3, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R2. **General:** Corrected typographical, grammatical and/or editorial errors. **Cover Page:** The Enforcement and Investigations Branch Chief was changed from Archie Lee to Acting Chief John Deatrick. The Ecological Assessment Branch Chief was changed from Bill Cosgrove to Acting Chief Mike Bowden. The FQM was changed from Liza Montalvo to Bobby Lewis. **Revision History:** Changes were made to reflect the current practice of only including the most recent changes in the revision history.	July 3, 2014
SESDPROC-202-R2, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R1.	October 15, 2010
SESDPROC-202-R1, *Management of Investigation Derived Waste*, replaces SESDPROC-202-R0.	November 1, 2007
SESDPROC-202-R0, Management of Investigation Derived Waste, Original Issue	February 05, 2007

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TABLES

Contents

1 General Information

1.1 Purpose

This document describes general and specific procedures and considerations to be used and observed when managing investigation derived waste (IDW) generated during the course of hazardous waste site investigations.

1.2 Scope/Application

The procedures and management options for the different categories of IDW described in this document are to be used by SESD field personnel to manage IDW generated during site investigations. On the occasion that SESD field personnel determine that any of the procedures described in this section are inappropriate, inadequate or impractical and that another procedure must be used to manage IDW generated at a particular site, the variant procedure will be documented in the field logbook, along with a description of the circumstances requiring its use. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

1.3 Documentation/Verification

This procedure was prepared by persons deemed technically competent by SESD management, based on their knowledge, skills and abilities and have been tested in practice and reviewed in print by a subject matter expert. The official copy of this procedure resides on the SESD Local Area Network (LAN). The Document Control Coordinator (DCC) is responsible for ensuring the most recent version of the procedure is placed on the LAN and for maintaining records of review conducted prior to its issuance.

1.4 References

SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, Most Recent Version

United States Environmental Protection Agency (US EPA). 2001. Environmental Investigations Standard Operating Procedures and Quality Assurance Manual. Region 4 Science and Ecosystem Support Division (SESD), Athens, GA

US EPA. Safety, Health and Environmental Management Program Procedures and Policy Manual. Region 4 SESD, Athens, GA, Most Recent Version

1.5 General Precautions

1.5.1 Safety

Proper safety precautions must be observed when managing IDW. Refer to the SESD Safety, Health and Environmental Management Program (SHEMP) Procedures and Policy Manual and any pertinent site-specific Health and Safety Plans (HASP) for guidelines on safety precautions. These guidelines, however, should only be used to complement the judgment of an experienced professional. Address chemicals that pose specific toxicity or safety concerns and follow any other relevant requirements, as appropriate.

1.5.2 Procedural Precautions

The following precautions should be considered when managing IDW:

- Due to time limitations and restrictions posed by RCRA regulations on storage of hazardous waste, accumulation start dates should be identified on all drums, buckets or other containers used to hold IDW so that it can be managed in a timely manner.
- During generation of both non-hazardous and hazardous IDW, keep hazardous IDW segregated from non-hazardous IDW to minimize the volume of hazardous IDW that must be properly managed.

2 Types of Investigation Derived Waste

Materials which may become IDW include, but are not limited to:

- Personal protective equipment (PPE) - This includes disposable coveralls, gloves, booties, respirator canisters, splash suits, etc.

- Disposable equipment and items - This includes plastic ground and equipment covers, aluminum foil, conduit pipe, composite liquid waste samplers (COLIWASAs), Teflon® tubing, broken or unused sample containers, sample container boxes, tape, etc.

- Soil cuttings from drilling or hand augering.

- Drilling mud or water used for mud or water rotary drilling.

- Groundwater obtained through well development or well purging.

- Cleaning fluids such as spent solvents and wash water.

- Packing and shipping materials.

Table 1, found at the end of this procedure, lists the types of IDW commonly generated during field investigations and the current disposal practices for these materials.

For the purpose of determining the ultimate disposition of IDW, it is typically distinguished as being either hazardous or non-hazardous. This determination is based on either clear regulatory guidance or by subsequent analysis. This determination and subsequent management is the responsibility of the program site manager.

3 Management of Non-Hazardous IDW

Disposal of non-hazardous IDW should be addressed in the study plan or QAPP for the investigation. To reduce the volume of any IDW transported back to the Field Equipment Center (FEC), it may be necessary to compact the waste into a reusable container, such as a 55-gallon drum.

If the waste is from an active facility, permission should be sought from the operator of the facility to place the non-hazardous PPE, disposable equipment, and/or paper/cardboard into the facility's dumpsters. If necessary, these materials may be placed into municipal dumpsters, with the permission of the owner. These materials may also be taken to a nearby permitted landfill. On larger studies, waste hauling services may be obtained and a dumpster located at the study site.

Disposal of non-hazardous IDW such as drill cuttings, drilling mud, purge or development water, decontamination wash water, etc., should be specified in the approved study plan or QAPP. It is recommended that these materials be placed into a unit with an environmental permit, such as a landfill or sanitary sewer. These materials must not be placed into dumpsters. If the facility at which the study is being conducted is active, permission should be sought to place these types of IDW into the facility's treatment system. It may be feasible to spread drill cuttings around the borehole, or, if the well is temporary, to place the cuttings back into the borehole. Non-hazardous monitoring well purge or development water may also be poured onto the ground down gradient of the monitoring well when site conditions permit. Purge water from private potable wells which are in service may be discharged directly onto the ground surface.

The minimum requirements for this subsection are:

- Non-hazardous liquid and soil/sediment IDW may be placed on the ground or returned to the source if doing so does not endanger human health or the environment or violate federal or state regulations. Under no circumstances, however, should monitoring well purge water be placed back into the well from which it came.
- Soap and water decontamination fluids and rinsates of such cannot be placed in any water bodies and must be collected and returned to the FEC for disposition.
- The collection, handling and proposed disposal method must be specified in the approved study plan or QAPP.

4 Management of Hazardous IDW

Disposal of hazardous or suspected hazardous IDW must be specified in the approved study plan or QAPP for the study or investigation. Hazardous IDW must be disposed as specified in USEPA regulations. If appropriate, these wastes may be placed back in an active facility waste treatment system. These wastes may also be disposed in the source area from which they originated if doing so does not endanger human health or the environment.

If on-site disposal is not feasible, and if the wastes are suspected to be hazardous, appropriate tests must be conducted to make that determination. If they are determined to be hazardous wastes, they must be properly contained and labeled. They may be stored on the site for a maximum of 90 days before they must be manifested and shipped to a permitted treatment or disposal facility. Generation of hazardous IDW must be anticipated, if possible, to allow arrangements for proper containerization, labeling, transportation and disposal/treatment in accordance with USEPA regulations.

The generation of hazardous IDW should be minimized to conserve Division resources. Most routine studies should not produce any hazardous IDW, with the possible exception of spent solvents and, possibly, purged groundwater. The use of solvents during field cleaning of equipment should be minimized by using solvent-free cleaning procedures for routine cleaning and decontamination (see SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205). If solvents are needed, the volume should be minimized by using only the amount necessary and by capturing the residual solvent separately from the aqueous decontamination fluids (detergent/wash water mixes and water rinses).

At a minimum, the requirements of the management of hazardous IDW are as follows:

- Spent solvents must be left on-site with the permission of site operator and proper disposal arranged.
- All hazardous IDW must be containerized. Proper handling and disposal should be arranged prior to commencement of field activities.

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Table 1: Disposal of IDW

TYPE	HAZARDOUS	NON - HAZARDOUS
PPE-Disposable	Containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.
PPE-Reusable	Decontaminate as per SESD Operating Procedure for Field Equipment Cleaning and Decontamination, SESDPROC-205, if possible. If the equipment cannot be decontaminated, containerize in plastic 5-gallon bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise return to FEC for proper disposal.	Decontaminate as per SESDPROC-205, and return to FEC.
Spent Solvents	Containerize in original containers. Clearly identify contents. Leave on-site with permission of site operator and arrange for proper disposal.	N/A
Soil Cuttings	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in a 55-gallon steel drum with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Groundwater	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. **
Decontamination Water	Containerize in DOT-approved container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. Decontamination water may also be disposed in a sanitary sewer system, with permission from the wastewater treatment plant representative, and if doing so does not endanger human health or the environment, or violate federal or state regulations.
Disposable Equipment	Containerize in DOT-approved container or 5-gallon plastic bucket with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal.	Containerize in an appropriate container with tight-fitting lid. Identify and leave on-site with permission of site operator, otherwise arrange with program site manager for testing and disposal. If unfeasible, return to FEC for disposal in dumpster.
Trash	N/A	Place waste in trash bag. Place in dumpster with permission of site operator, otherwise return to FEC for disposal in dumpster.

**** These materials may be placed on the ground if doing so does not endanger human health or the environment or violate federal or state regulations.**

SUBSAMPLING AND COMPOSITING OF SAMPLES

DOCUMENT I.D. SOILPREP-SUBS

Approved By: ______________________ Date: 8/8/18
Sample Preparation Manager, Jonathon Walter

Prepared By: ______________________ Date: 8/6/18
Quality Assurance Manager, Carl Degner

Prepared By: ______________________ Date: 8/9/18
General Manager, Ambrose Hughey

Annual Review:

Reviewed By: ______________________ Date: ____________

Reviewed By: ______________________ Date: ____________

Reviewed By: ______________________ Date: ____________

Reviewed By: ______________________ Date: ____________

Reviewed By: ______________________ Date: ____________

Doc Control ID:	____________	Archived Date:	____________

ALS-Kelso SOP Annual Review Statement

SOP Code: SOILPREP-SUBS
Revision: 1

An annual review of the SOP listed was completed on (date): **7/13/18**

☐ The SOP reflects current practices and requires no procedural changes.

Supervisor: Date:

☒ Revision of the SOP is needed to reflect current practices. Draft revisions are listed below.

SOP Section Number	**Description of Revision Needed**	**Date Procedure Change Implemented**	**Supervisor Initials** Indicating Approval of Revision
11.7.2	When creating aliquots for METALS, Mercury aliquots should be 5 grams and all other metals aliquots should be 10 grams.	5/30/17	SC
	Current Templates with locked calculation cells to be added to the SOP.	2/1/18	SC
11.2	Remove all of this section on aqueos samples.	7/16/18	SC
11.10.2	Remove	7/16/18	SC
11.10.3	Remove	7/16/18	SC

ALS-Kelso SOP Annual Review Statement

SOP Code: SOILPREP-SUBS

Revision: 1

11.10.4	Remove	7/16/18	SC
11.10.5	Third bullet point should read "Cut strips 7 1/2, 5, and 2 1/2 inches. This will make a total of 16 2 1/2" squares. The fourth bullet point should read place all squares into a large ziploc bag.	7/16/18	SC
11.10.7	Should read " Determine the total mass of sample neeed to aliquot for all tests. Divide the total mass by the number of samples to be composited. Shed that mass of each sample to be composited together into a single container. Homogenize throughly once all fractions have been shredded. Aliquot each sub sample from this final mass."	7/16/18	SC
11.10.8	Remove	7/16/18	SC
11.10.9	Remove	7/16/18	SC
11.3.4	Add section: Air drying: If samples are to be air dried they will be set out in cleaned pans to dry at room temp and conditions. If the samples are for DOD work, they will be dried to a constant weight with a confirmation of 4% or less weight loss. The sample weights will be recorded on the constant weight bench sheet.	8/23/18	SC/JW

Attach additional pages or information if necessary

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1) Scope & Applicability

1.1 This standard operating procedure describes procedures for obtaining subsamples used for laboratory analysis. The procedure also describes general practices for making composite samples from multiple individual samples. Procedures are given for aqueous, soil, sediment, vegetation and miscellaneous matrices. The SOP does not apply to tissue samples. Procedures for tissue samples are described in the GEN-TISP and MET-TDIG SOPs.

1.2 The SOP describes routine, or default, procedures for samples that do not require VOC analyses. Handling of VOC samples is described in SOP VOC-5035. Program or project-specific requirements may differ from those described in the SOP. Samples analyzed by EPA CLP procedures are specifically excluded from this procedure, and will be handled according to the applicable SOW.

1.3 Multi-increment samples require special handling and subsampling procedures. In addition to routine procedures, this SOP also includes instructions for handling and sampling from multi-increment samples submitted to the laboratory.

1.4 This procedure does not apply to situations where the entire sample (container) is used for the analysis.

1.5 In cases where there is a project-specific quality assurance plan (QAPP), the project manager identifies and communicates the QAPP-specific requirements to the laboratory. In general, project specific QAPP's supersede method specified requirements. For example, projects falling under DOD ELAP. QC requirements defined in the SOP *Department of Defense Projects – Laboratory Practices and Project Management (ADM-DOD/ADM-DOD5)* may supersede the requirements defined in this SOP,

2) Summary of Procedure

2.1 Obtaining a representative analytical subsample from the field sample submitted is essential to providing meaningful data. The subsample must be taken to most closely reflect the predominant composition of the sample. For aqueous and liquid samples, this is usually accomplished by shaking or inverting the sample. For soil, sediment, powders, and other solids the procedures are more involved. Procedures for subsampling are based on the information given in the references listed.

2.2 Some projects may employ multi-increment (MI) sampling in the field. The primary objective of MI sampling is to control the certain statistical errors associated with discrete sampling. Some studies have shown that MI sampling, using 30+ sample increments within a decision unit (a defined field sampling area) may provide a more representative view of contaminant concentrations than traditional discrete sampling approaches. References listed provide additional background on MI sampling. When this approach is taken it is important that laboratory procedures are consistent with field procedures when taking samples.

2.3 Unique sample matrices such as vegetation, wood and wood chips, mechanical parts and filters, etc. pose additional challenges to obtaining representative samples. For these samples the laboratory staff should consult with the Project Manager to determine the subsampling strategy. These special situations will be handled on a case-by-case basis. Service requests should list any specific sample preparation required.

3) Definitions

3.1 Batch - A batch of samples is a group of environmental samples that are prepared and/or analyzed together as a unit with the same process and personnel using the same lot(s) of reagents. It is the basic unit for analytical quality control.

3.1.1 Preparation Batch - A preparation batch is composed of one to twenty field samples, all of the same matrix, and with a maximum time between the start of processing of the first and last samples in the batch to be 24 hours.

3.1.2 Analysis Batch - Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration (initial or continuing verification) followed by sample extracts interspersed with calibration standards (CCBs, CCVs, etc.) The sequence ends when the set of samples has been injected or when qualitative and/or quantitative QC criteria indicate an out-of-control situation.

3.2 Sample

3.2.1 Field Sample - An environmental sample collected and delivered to the laboratory for analysis; a.k.a., client's sample.

3.2.2 Laboratory Sample - A representative portion, aliquot, or subsample of a field sample upon which laboratory analyses are made and results generated.

3.2.3 Sample – A portion of material taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity (ASTM).

3.2.4 Subsample – A portion of a sample taken for the purpose of estimating properties or composition of the whole sample (ASTM).

3.2.5 Composite sample – A mixture of multiple samples or subsamples produced to result in one sample representative of multiple field samples.

3.2.6 Representative subsample – A subsample collected in such a manner that it reflects one or more characteristics of interest (a defined by the project objectives) of the laboratory sample from which it was collected (ASTM).

3.2.7 Multilayered sample – A sample consisting of two or more clearly differentiated components (ASTM).

3.2.8 Multi-increment sample (MIS) – A field sample consisting of multiple bulk containers from one decision unit (defined in a MIS sampling plan) submitted to the lab for subsampling into a representative sample for analysis. Also known as Incremental Sampling Methodology (ISM).

3.3 Method Blank (MB) - The method blank is an artificial sample composed of analyte-free water or solid matrix and is designed to monitor the introduction of artifacts into the analytical process. The method blank is carried through the entire analytical procedure.

3.4 Laboratory Control Samples (LCS) – The LCS is an aliquot of analyte free water or analyte free solid to which known amounts of target analytes are added. The LCS is prepared and analyzed in exactly the same manner as the samples. The percent recovery is compared to established limits and assists in determining whether the batch is in control.

3.5 Duplicates and Duplicate Matrix Spikes are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed.

4) Responsibilities

4.1 It is the responsibility of the analyst to perform the analysis according to this SOP and to complete all documentation required for data review. Analysis and interpretation of the results are performed by personnel in the laboratory who have demonstrated the ability to generate acceptable results utilizing this SOP. This demonstration is in accordance with the training program of the laboratory. The department supervisor/manager or designee performs final review and sign-off of the data.

4.2 It is the responsibility of the department supervisor/manager to document analyst training and method proficiency, as described in the *ALS-Kelso Training Procedure* (ADM-TRAIN).

5) Interferences

5.1 When obtaining subsamples it is important to minimize any chances for sample contamination or cross-contamination between samples. Work should be performed in an organized and neat manner. Spilling of samples (from overfilled containers, etc.) should be minimized and spills cleaned up. Equipment and laboratory tools used with samples should be cleaned between samples to prevent cross-contamination.

5.2 Analysis-specific interferences are described in the applicable analytical SOP.

6) Safety

6.1 All appropriate safety precautions for handling solvents, reagents and samples must be taken when performing this procedure. This includes the use of personal protective equipment, such as, safety glasses, lab coat and the correct gloves.

6.2 Chemicals, reagents and standards must be handled as described in the ALS safety policies, approved methods and in SDSs where available. Refer to the ALS Chemical Hygiene Plan and the appropriate SDSs prior to beginning this method.

7) Sample Collection, Containers, Preservation, and Storage

7.1 Refer to the analytical SOP for sample collection preservation and storage of samples. Subsamples and composite samples held for later analysis should be preserved and stored in the same manner as specified for field samples.

7.2 MIS Projects

7.2.1 Projects for MI samples may include additional instructions not found in the analytical SOP. The analyst should consult with the Project Manager, or refer to the Project Manager's instructions, prior to working with these samples.

7.2.2 LIMS test codes are used to specify which MIS-analytical tests are needed (e.g. ISM-PAH). These test codes will have holding times associated with them that will ensure the completion of the MIS work before the initial analytical holding times (e.g. sample extraction) lapse.

8) Apparatus and Equipment

8.1 Laboratory balance capable of weighing the desired sample mass. There are various makes and models of balances available for use, with each department having balances appropriate for its use. For weighing solids and non-aqueous liquids (wastes), use a top-loader balance. Ensure that the mass (sample + container) to be placed on the pan is within the calibration-verified range of the balance.

8.2 Balance calibration verifications must be performed prior to use on each day of use. The calibration verification weights must bracket the range of use. For additional information, refer to the SOP *Documenting Laboratory Balance and Temperature Checks* (ADM-BAL).

8.3 Wiley laboratory mill, Model 4. Operate the Wiley mill following the manufacturer's recommendations.

8.4 Sieve shakers.

8.5 Shatter box.

8.6 Mechanical mixer and/or shaker.

8.7 Stainless steel or Glass mixing bowl.

8.8 Metal or disposable spoons and spatulas.

8.9 Aluminum foil.

8.10 Weighing boats, plastic or aluminum.

8.11 Clean sample containers and lids (various sizes) as specified in the applicable test SOP.

8.12 Common laboratory glassware/apparatus (beakers, flasks, pipets, syringes, etc.).

8.13 Multi-Increment Samples

8.13.1 Flat spatula, modified to create sides perpendicular to the flat surface used to scoop.

8.13.2 Flat stainless steel masons trowel.

8.13.3 Volatile sample containers.

8.13.3.1 250-500 milliliter (mL) narrow mouth, amber bottles (recommended).

8.13.3.2 4-8 ounce (oz.) amber jars with Teflon lined septum lids.

8.13.4 Large stainless steel spoon or scoop.

8.13.5 Large clean containers (a large stainless steel or glass bowl, Ziploc bags, or 5 gallon bucket).

8.13.6 #10 (2 mm) sieve.

8.13.7 Stainless steel cookie sheet or other tray.

9) Standards, Reagents, and Consumable Materials

9.1 All stocks, working solutions and sample dilutions should be prepared using deionized water (DI) conforming to ASTM Type I or ASTM Type II reagent water. For more information on reagent water generation, refer to the related SOP, Operation and Maintenance of Laboratory Reagent Water Systems.

9.2 To provide traceability, manufacturer lot numbers of solvents, reagents, standards and supplies used in an analysis shall be recorded on each analytical procedure's batch record, whether it is on the analytical record and/or into a logbook.

9.3 Dichloromethane, acetone, methanol, and acetonitrile may be used during the noted procedures for cleaning and decontamination of equipment.

10) Preventive Maintenance

10.1 All maintenance activities are recorded in a maintenance logbook kept for each instrument. Pertinent information (serial numbers, instrument I.D., etc.) must be in the logbook. This includes the routine maintenance described herein. The entry in the log must include: date of event, the initials of who performed the work, and a reference to return to analytical control.

11) Procedure

11.1 Aqueous samples - Subsampling

11.1.1 Examine the sample. Thoroughly mix all samples by vigorous shaking. Immediately open the container and obtain the subsample. Additional filtering of the subsample may be required by the analytical SOP.

11.1.2 If the sample is multi-layered (a water layer with a sand/sediment layer that cannot be mixed or non-aqueous liquid layer) the Project Manager should be consulted on how to proceed with the sample. Additional analyses or sample preparations may be necessary depending on the client's data needs. Document the condition of the sample and decision made on subsampling.

11.2 General considerations – Non-liquid samples

11.2.1 The analyst must first understand what the sample matrix of interest is. The project information should be consulted. If the sample appears to be homogeneous (other than extraneous materials described below) particle size reduction is not necessary. Particle size reduction should be performed only when required by the project QAPP, project specifications, or client request. If particle size reduction is required, use the appropriate apparatus (Wiley mill, shatter box, etc.) to perform crushing, grinding, milling, or sieving, and document. Refer to ASTM D6323 for guidelines on performing particle size reduction.

11.2.2 Once the matrix of interest is known, examine the sample for presence of extraneous material. The default procedure is to remove these items, or not include in the representative subsample. However, the presence of these materials should be documented in lab records and the Project Manager should be consulted prior to subsampling. Some examples are given below.

- Soil, solid, and sediment samples may include such material as larger, rocks, sticks, leaves, pieces of metal, man-made materials, etc.
- Wood or bark samples may include chunks of soil, mud, rocks, etc.
- Vegetation samples may include chunks of soil, mud, rocks, sticks (not of the sample type, etc.).
- Sediment samples may include rocks, twigs, vegetation, organisms, etc.
- Sediment/marine projects, organisms are typically analyzed under separate sampling and analysis plans.
- Mechanical parts, filters, etc., may include chunks of soil, mud, rocks, sticks, etc.

11.2.3 Examine soil samples to determine if the sample contains significant amounts of water. If the amount of water is greater than approximately 30%, treat the sample as a sediment sample.

11.2.4 Samples which are especially heterogeneous, as well as various special matrices, may require additional preparation. These will be handled on a case-by-case basis after consultation with the appropriate supervisors and Project Manager. Unique matrices for TCLP and other leaching procedures should be handled according to the applicable SOP or reference method.

11.3 Soil/solid Samples

11.3.1 Subsampling samples in jars

11.3.1.1 Using a spatula or other utensil made of an inert material, thoroughly mix and homogenize the sample, making sure to loosen sample from the sides of the container, and continue mixing the entire contents, breaking up soil clumps, etc., until there is no visible segregation of the sample by layer, grain size, color, etc. The sample should appear uniform in color and texture.

11.3.1.2 Once mixed, remove the desired mass of sample for the analysis and document accordingly. Recap the jar and return to storage.

11.3.2 Subsampling samples in sleeves (core samples) and large bulk containers.

11.3.2.1 Empty samples in sleeves into a metal or glass homogenizing container and thoroughly stir using a spatula or other utensil. When homogenized the appropriate sample portions are placed in jars. Perform additional drying and grinding only when specified for the project. Client specifications for drying and grinding will be communicated by the Project Manager.

11.3.2.2 When working with sleeves and resulting homogenized samples or subsamples, always double-check the sample ID on the sleeve against the sample numbers on the samples.

11.3.3 Compositing soil/solid samples

11.3.3.1 Thoroughly mix each individual sample as described above.

11.3.3.2 Combine equal masses from each of the individual samples into a clean stainless steel mixing bowl. The amount used will depend upon the number of analyses to be performed on the composite and/or the amount available. The analyst preparing the composite will document the mass of each individual sample used for the composite, the date and time of compositing, and any other pertinent observations using the Composite Data benchsheet.

11.3.3.3 Thoroughly homogenize the sample using a spatula or other utensil and returned to clean glass jars. The sample container is labeled as a composite and with the sample identification.

11.3.3.4 Return the composite sample and remaining individual samples to storage.

11.4 Sediment Samples - Subsampling

11.4.1 Standard procedure calls for mixing overlying water into the sample. EPA SW-846 methods for organic extractions specify to decant and discard overlying water. However, the Puget Sound Protocols and others have options for decanting and discarding this water, decanting and performing a separate water analysis, or mixing the water into the sample. The analyst should confirm which option is to be used on the sample. For projects not within the scope of the Puget Sound Protocols or similar project plans, the overlying water should be decanted and discarded for organics analysis. For metals and inorganics, mix the overlying water into the sample.

Note: If water is decanted and discarded and percent solids is to be applied or determined, a separate solids determination must be made on the decanted sample.

11.4.2 Thoroughly mix and homogenize the sample, making sure to mix the entire contents of the jar. Additional steps may be needed to homogenize the sample (break up soil clumps, etc.). The sample should be mixed so there is a uniform color and texture.

Note: Sediment samples may contain considerable amounts of organics matter. Ensure that samples are thoroughly mixed. Document the presence of substantial organic matter, shells, etc.

11.4.3 Once mixed, remove the desired mass of sample for the analysis and document accordingly. Recap the jar and return to storage.

11.4.4 The subsample is transferred to an appropriate, labeled container. The sample container is stored in the appropriate refrigerator in sample receiving and any empty sleeve can be stored at room temperature.

11.5 Sediment Samples - Compositing

11.5.1 Thoroughly mix each individual sample as described above.

11.5.2 Combine equal masses from each of the individual samples into a clean stainless steel or glass mixing bowl. The amount used will depend upon the number of analyses to be performed on the composite and/or the amount available. The analyst preparing the composite will document the mass of each individual sample used for the composite, the date and time of compositing, and any other pertinent observations using the Composite Data benchsheet.

Note: Equal masses are used unless otherwise instructed. It may be required to use the entire jar or other measure.

11.5.3 The sample is thoroughly homogenized using a spatula or other utensil and returned to clean glass jars. The sample container is labeled as a composite and with the sample identification, dated, and initialed.

11.5.4 The composite sample and remaining individual samples are returned to storage.

11.5.5 Samples should be received prepared from the field as sample increments.

Although unlikely, in cases where proper preparation of increments from large bulk samples does not occur in the field, the following steps will be taken.

11.5.5.1 When obtaining sample increments from a large bulk container (bucket, large jar, large bag, etc.) be sure to sample from the center and remove the soil 1-2 inches deep. Using the large spoon or scoop, collect the sample increment according to the work plan. Scoop approximately 30-60 grams into a large, clean container and move on to the next sample increment location. Be cautious of oversize material, which means more mass may need to be taken from each increment to end with the 30–50 g sub-sample after sieving (a 5 Kg field sample may not be uncommon). Increments can be sieved directly into the bucket, or they can be bagged and sieved later.

11.6 Multi-Incremental Sampling (or Incremental Sampling Methodology (ISM)) – When laboratory subsampling using MIS/ISM is to be used to produce the analytical subsample(s), the following procedures are used. If, after reviewing the project and Service Request information, the analyst has any uncertainty of the MIS approach to take, they must confirm with the Project Manager the protocol to be used.

NOTE: The default procedure is to be used when no other client or project specifications or modifications are given. This section refers to two tables – one specifying default increment amounts for analytical and one listing a "large mass" option that is to be used only when project specified. In addition, the State of Hawaii DOH protocol is to be used when requested. A procedure for the analysis by Method 8330B is also given.

11.6.1 Default procedure

11.6.1.1 After the 30-50 sample increments have been field collected into a container (a 5 Kg field sample may not be uncommon) air dry the entire sample (all received containers) in aluminum pans pre-rinsed 3 times with DCM (dichloromethane/methylene chloride). Note, if Aluminum is a target analyte of interest then substitute the aluminum pans for glass or stainless steel. Air drying may take 2-4 days with occasional stirring.

11.6.1.2 The intent of air drying is to convert the sample to a more manageable form prior to sieving. The sample is considered air-dried when the material appears dry enough to enable disaggregation and sieving. Due to high variability of laboratory samples, sample dryness should be confirmed by a senior analyst or supervisor prior to going further with the procedure. For work performed under DOD QSM, constant weight data will be recorded on the Constant Weight Data Sheet (Hyperlink in Section 20).

11.6.1.3 Rinse all utensils and equipment with DCM three times prior to use (stainless steel tray, mortar & pestle, 2 mm sieve & catch pan, trowel, ISM spatula).

11.6.1.4 Lightly grind the air dried sample with a mortar & pestle in order to break up dirt and clay chunks (do not size reduce rocks or vegetation) and pass sample through a 2 mm sieve.

11.6.1.5 Weigh the remaining +2 mm fraction in an appropriate sized jar and record the weight on the Air Dried Sieve Data benchsheet (Figure 1). Describe the +2mm fraction on the bench sheet (size of rocks, type of any vegetation, etc.).

11.6.1.6 Weigh and record the weight of the -2 mm fraction on the Air Dried Sieve Data benchsheet (Figure 1).

11.6.1.7 Mix the sample, dump on a DCM-rinsed stainless steel pan, and spread the sample out with a trowel, forming a rectangle no more than 1cm deep.

11.6.1.8 Divide the sample into a minimum of 30 equal sections (30 to 50 sections is recommended) using the trowel blade. Note that the entire sample should be included in the grid and amount of sample ‘outside’ the grid outer edges minimized (however, do not overly manipulate the sample in an attempt to create a perfect grid).

11.6.1.8.1 Collect an equal (approximate) amount of sample from each of the sections based on the applicable table (Table 1 or Table 2) and place into a labeled container (see Tables 1 and 2). Scrape the modified flat spatula along the bottom of the tray and pull straight up to make sure all depths and particle sizes are represented in the collection area. Avoid collecting portions from the edge of gridlines (where the slab has been disturbed). Record the exact final weight of sample for each test on the ISM bench sheet and on the jar. All weights shall be measured using an analytical balance.

11.6.1.8.2 Since each laboratory area must analyze the entire contents of the prepared (or submitted) jar, the subsampling process must be repeated for each separate analysis to be performed on the sample. The subsampling process must be performed for each individual QC sample as well. The entire mass in the jar will be analyzed (TOC is the exception). The results may be less defensible if only a subsample or fraction of the jar contents is analyzed.

11.6.1.8.3 If sample amount is sufficient, it is recommended to repeat the process to obtain a backup sample in the event that re-analysis is required. This ‘As Received’ backup is placed back in the original sample jar and returned to sample management/custody.

11.6.1.9 Labeling and storage

11.6.1.9.1 Refer to Table 3 for default storage conditions, which are based on how the MIS sample was prepared and on the stability/volatility of target analytes.

11.6.1.9.2 MIS subsamples do not need to be returned to SMO for barcode labeling. Label the aliquots with labels from "prep App" and deliver them directly to the labs. Document the internal custody transfer directly on the benchsheet that is delivered with the samples.

11.6.1.9.3 Place any remaining -2mm sample into jars labeled as "-2 mm archive." If there are multiple jars, label them as "1 of 3", "2 of 3", etc. All remaining bulk sample jars must be returned to SMO for barcode labeling and storage.

11.6.1.9.4 Usually, the -2 mm archive and test archive (back-up samples) jars are placed in a freezer, while the +2 mm archive and test jars (with QC) are placed on the room temperature shelves.

11.6.2 Procedure for ISM following State of Hawaii DOH Protocol (see references)

11.6.2.1 Samples requesting the Hawaii DOH procedure require wet and/or dry sieving depending on the test/analytes for which subsamples are being prepared. Refer to a copy of the Hawaii DOH procedure and/or the Project Manager for details before beginning.

11.6.2.2 Obtain instructions from the Project Manager or Service Request for increment amounts and test subsample amounts. Also refer to the *Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan*, November 12, 2008, Section 4.2.2 for guidance on increment/sample amounts.

11.6.2.3 Subsample bulk MI samples to be tested for SVOCs, including TPH-D, some PAHs, Mercury, and unstable pesticides, should be subsampled without drying or sieving in order to minimize chemical loss or alteration and meet holding times for analysis. Refer to Table 2a of *Technical Guidance Manual Notes: Decision Unit and Multi-Increment Sample Investigations*, March 2011, State of Hawaii, Department of Health, and Reference document number 2011-143-RB.

11.6.2.4 If both SVOC and non-volatile PAHs are targeted contaminants of interest then include testing for both in laboratory subsamples collected from the multi-Increment sample prior to drying and sieving.

11.6.2.5 When creating aliquots for METALS, Mercury aliquots should be 5 grams and all other materials aliquots should be 10 grams.

11.6.2.6 For wet ISM aliquots, organic tests (SVG/SVM) require a larger aliquot size to accommodate for the extra water content. In most cases, low-level organic tests will require a 40 g wet aliquot (max weight capacity for most tests) and normal level tests will require a 20 g wet aliquot (double the target dry weight).

11.6.2.7 Use a separate sample from the wet material and test for soil moisture in order to convert analytical results to dry-weight basis.

11.6.2.8 Not all samples from Hawaii require the State of Hawaii DOH procedure. See service request and/or verify with the Project Manager.

11.6.3 Procedure for ISM on 8330B Explosives

11.6.3.1 Samples from Ammunition Depots and anywhere except Firing Ranges (not DOD).

11.6.3.1.1 Follow the basic ISM procedure, except all utensils/pans need rinsed 3 times with Acetonitrile (instead of DCM). Collect a 10.00 g aliquot and place in a 4 oz. amber jar (explosives are UV sensitive).

11.6.3.2 Samples from Firing Ranges

11.6.3.2.1 Grinding: For firing ranges, the entire -2 mm portion collected from the sieving procedure must be ground to a powder in the shatter box.

11.6.3.3 Method 8330B DOD samples

11.6.3.3.1 Grinding: For DOD work, the entire -2 mm portion collected from the sieving procedure <u>must</u> be ground to a powder in the shatter box prior to proceeding. Note: high-speed milling, such as in the shatter box, can elevate sample temperature due to friction. The thermal stability of the target analytes should be considered when performing this grinding procedure. Method 8330B specifies a 2 minute (or longer) cool down period between every five, 60 second grinding intervals to maintain acceptable temperatures and minimize loss of volatile energetic contaminants.

11.6.3.3.2 An SRM (supplied by the Organic LC instrument lab) must be taken through the grinding and ISM procedure (already dry so doesn't need to be air dried or sieved). Shatter box 50 - 100 g of the well-mixed SRM, and then make a 10 g aliquot after grinding. Place the aliquot in 4 oz. amber jar. Archive the remaining SRM in an amber jar.

11.6.3.3.3 Grinding Blank: Matrix sand blanks (use baked sand) must be ground in the shatter box between each sample and aliquoted following the ISM procedure. The blanks can be ground in equal portions and then recombined at the end to make one sample requiring one ISM aliquot procedure. (Example: To ISM a 200 g portion for use in making the final 10 g aliquot, divide 200 g by the number of samples needing shatter box and grind that amount of matrix sand between each sample. Recombine all ground matrix sand at the end and ISM one 10 g aliquot from the 200 g of ground matrix sand.) Archive the remaining matrix sand in an amber jar.

11.7 Analyte-Specific Considerations

11.7.1 Metals

11.7.1.1 It has been proven that grinding can greatly improve the reproducibility for metals analyses. However, erosion of the metals surfaces used in grinding may contribute to a high bias in the samples. It is recommended that the tungsten carbide grinding mill is used when grinding soils in the shatter box thereby limiting the amount of potential bias in the prepared samples.

11.7.1.2 When grinding soil samples that may potentially contain ores of malleable metals (e.g. Lead, Copper, Tin) be aware that the malleable particles may tend to smear during grinding, and may be lost from the samples to equipment surfaces. This anomaly may bias sample results low, decontamination of equipment surfaces may be difficult and could result in high bias in subsequent samples from carry over.

11.7.1.3 Reproducibility for Lead analyses in unground, incrementally sampled (IS) samples from small arms firing ranges may have an unacceptable large variability. The large variability for Lead may be due to single particles of Lead between one and two millimeters in diameter being present in only some of the replicate splits. If the end data is to assess risk of accidental ingestion of Lead, precision for the concentration of lead contained in larger particles may be of less interest then the Lead contained in the finer, less than 0.25 mm, fraction. Using a finer mesh sieve (0.25 mm rather than 2 mm) may improve precision and reproducibility. However, sieving unground samples through sieves finer than two millimeters is not appropriate if analyzing for high explosives or propellants. Typical mass sizes for energetic analytes are in particles sizes greater than 0.59 millimeters.

11.7.1.4 MI samples collected for Arsenic analyses that contain greater than 20 mg/Kg total Arsenic should be tested for bioaccessible Arsenic. This should be discussed with the project manager. If deemed appropriate, the entire <2 mm fraction of the respective samples should be sieved to a ≤0.25 mm, representatively sub-sampled and analyzed for bioaccessible Arsenic using SBRC methodology, 1-2 grams are required.

11.7.2 Polycyclic Aromatic Hydrocarbons (PAHs)

11.7.2.1 Currently there is little information in published procedures specific to the laboratory processing of ISM samples for PAHs. The default procedure above is used, but the 8330B procedure is an acceptable option if specified.

11.7.3 Perchlorate

11.7.3.1 Currently there is little information in published procedures specific to the laboratory processing of ISM samples for Perchlorate. Laboratory processing of samples per EPA Method 8330B as described in Section 11.7.3 is recommended. A 10 gram sample is required for propellants and explosives. It is recommended that a 10 gram ISM sample should be extracted with 100 mL of DI water for Perchlorate analysis by EPA Method 314.0.

11.8 Vegetation samples

11.8.1 Since vegetation samples often are not amenable to standard mixing and homogenization techniques, or because specific sections of the vegetation are targeted, these are handled on a case-by-case basis with instructions from the Project Manager. The PM will obtain sample-specific instructions from the client, and then communicate the specifications to the lab personnel using the ALS Form V or similar project specification document for the project. If the client makes reference to specific procedures, methods, or technical references, the PM will make the document(s) available to the laboratory personnel.

11.9 Paperboard samples

11.9.1 In general, prepare paperboard samples as described below. Project-specific instructions may replace these.

11.9.2 Prepare the FDA Ext first.

- Cut the sheet of paper into one 10” x 10” square.
- Cut the 10” x 10” into strips at the cut lines 7 ½, 5, and 2 ½.
- Cut strips at 7 ½, 5, and 2 ½ inches. This will make a total of 16, 2½” squares.
- Place all the squares into a large Zip Lock™ bag.

11.9.3 Put one sheet of paper into shredder, run the shredder back and forth to get the entire sample out. Use tongs to remove any remaining sample in bottom of shredder. As a safety precaution, ensure the unit is in the “OFF” position.

11.9.4 Determine the total mass of sample needed to aliquot for all tests. Divide the total mass by the number of samples to be composited. Shred that mass of each sample to be composited together into a single container. Homogenize thoroughly once all the fractions have been shredded. Aliquot each sub sample from this final mass.

11.9.5 Update composites as being done. Open StarLIMS, double click on Ad Hoc by Test (Under Results entry), highlight samples composited and click the Update to Done button at the top of page. Do not add jars when asked. Just click the X on the right hand corner.

12) Quality Assurance/Quality Control Requirements

12.1 This method shall operate under the formal Quality Assurance Program established at ALS and must maintain records that define the quality of data that is generated. Data shall be compared to established criteria in order to determine if the results meet the performance characteristics of the determinative method(s).

12.2 Ongoing QC Samples required for each sample batch (20 or fewer samples) are described in the SOP for Sample Batches and in the determinative SOPs.

13) Data Reduction and Reporting

13.1 All compositing and subsampling data must be recorded into the bench records by the analyst. In addition to sample volumes and masses, sample identifications, etc., this should include descriptions of unique samples or sample components. Figure 1 shows the current MIS benchsheet template used to record MIS subsampling. Other project-specific benchsheets may apply.

13.2 It is the supervisor's responsibility to ensure that analytical data is reviewed and to ensure that all quality control requirements have been met.

14) Method Performance

14.1 Not applicable.

15) Pollution Prevention and Waste Management

15.1 It is the laboratory's practice to minimize the amount of solvents, acids and reagent used to perform this method wherever feasible. Standards are prepared in volumes consistent with methodology and only the amount needed for routine laboratory use is kept on site. The threat to the environment from solvent and reagents used in this method can be minimized when recycled or disposed of properly.

15.2 The laboratory will comply with all Federal, State and local regulations governing waste management, particularly the hazardous waste identification rules and land disposal restrictions as specified in the ALS Lab Waste Management Plan.

15.3 This method uses non-halogenated solvents and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and disposed of in accordance with Federal and State regulations.

15.4 This method uses Dichloromethane and any waste generated from this solvent must be placed in the collection cans in the lab. The solvent will then be added to the hazardous waste storage area and recycled off site.

16) Corrective Actions for Out-of-Control Data or Unacceptable Data

16.1 Refer to the SOP for *Non Conformance and Corrective Action* (CE-QA008) for procedures for corrective action. Personnel at all levels and positions in the laboratory are to be alert to identifying problems and nonconformities when errors, deficiencies, or out-of-control situations are detected.

16.2 Handling out-of-control or unacceptable data

16.2.1 On-the-spot corrective actions that are routinely made by analysts and result in acceptable analyses should be documented as normal operating procedures, and no specific documentation need be made other than notations in laboratory maintenance logbooks, run logs, for example.

16.2.2 Some examples when documentation of a nonconformity is required using a Nonconformity and Corrective Action Report (NCAR):

- Quality control results outside acceptance limits for accuracy and precision.
- Method blanks or continuing calibration blanks (CCBs) with target analytes above acceptable levels.
- Sample holding time missed due to laboratory error or operations.
- Deviations from SOPs or project requirements.
- Laboratory analysis errors impacting sample or QC results.
- Miscellaneous laboratory errors (spilled sample, incorrect spiking, etc).
- Sample preservation or handling discrepancies due to laboratory or operations error.

17) Training

17.1 Refer to the SOP *ALS-Kelso Training Procedure* (ADM-TRAIN).

17.2 Training outline

17.2.1 Review literature (see references section). Read and understand the SOP. Also review the applicable SDS for all reagents and standards used. Following these reviews, observe the procedure performed by an experienced analyst at least three times.

17.2.2 The next training step is to assist in the procedure under the guidance of an experienced analyst. During this period, the analyst is expected to transition from a role of assisting, to performing the procedure with minimal oversight from an experienced analyst.

17.3 Training is documented following the SOP *ALS-Kelso Training Procedure* (ADM-TRAIN).

NOTE: When the analyst training is documented by the supervisor on internal training documentation forms, the supervisor is acknowledging that the analyst has read and understands this SOP and that adequate training has been given to the analyst to competently perform the analysis independently.

18) Method Modifications

18.1 Not applicable.

19) Summary of Changes

19.1 Updated to the latest ALS SOP format.

19.2 Minor typographical, grammatical, and formatting revisions.

19.3 Signature Page: Updated signatories.

19.4 Section 11.6.2.5: Updated Metals Hg aliquot sampling.

19.5 Section 11: Numerous edits, additions, and changes to reflect current practice.

19.6 Section 20: Current templates with locked calculation cells added to the SOP.

20) References and Related Documents

20.1 Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples, U.S. Environmental Protection Agency, EPA/600/R-03/027, November 2003.

20.2 Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities, ASTM D 6323, Annual Book of ASTM Standards, 1999.

20.3 Test Methods for Evaluating Solid Waste, EPA SW-846, Final Update III, December 1996.

20.4 Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound, January, 1996.

20.5 Draft Guidance on Multi-Increment Soil Sampling State of Alaska, Department of Environmental Conservation, March 2007.

20.6 Technical Guidance Manual for the Implementation of the Hawaii State Contingency Plan, November 12, 2008.

20.7 Technical Guidance Manual Notes: Decision Unit and Multi-Increment Sample Investigations, March 2011, State of Hawaii, Department of Health, 2011-143-RB.

20.8 Standard operating Procedure, In Vitro Method for Determination of Lead and Arsenic Bioavailability; Solubility/Bioavailability Research Consortium, Document 8601-102.011- 0601-1099-RN01.

20.9 Figure 1: Multi Incremental Sampling Worksheet.

20.10 Analytical Worksheets:

20.10.1 Blank Bench Sheet: R:\Soil Prep\Templates\Blank Bench sheet REV1.xltx.
20.10.2 Constant Weights Data Sheet: R:\Soil Prep\Templates\Constant Weight Data Sheet REV1.xltx.
20.10.3 Foil Rinse Bench sheet: R:\Soil Prep\Templates\Foil Rinse Bench Sheet REV1.xlsx.

20.10.4 Paperboard Composite Data: R:\Soil Prep\Templates\Paperboard Composite Data REV2.xlsx.
20.10.5 Sieve Data Sheet: R:\Soil Prep\Templates\Sieve Data Sheet REV3.xltx.
20.10.6 Soil Composite Data Sheet: R:\Soil Prep\Templates\Soil Composite Data Sheet REV3.xltx.
20.10.7 Soil Grinding Data Sheet: R:\Soil Prep\Templates\Soil Grinding Data Sheet. REV2.xltx.

20.11 TNI Standard, Volume 1- 2009.

20.12 DoD Quality Systems Manual for Environmental Laboratories Version(s) 5.0/5.1.

21) Attachments/Appendices

21.1 Table 1: Default Multi – Incremental Sampling Information.

21.2 Table 2: Large Mass Multi – Incremental Sampling Information.

21.3 Table 3: Storage of Multi-Incremental Subsamples.

21.4 Figure 1: Air Dried Sieve Data Benchsheet Template.

TABLE 1
Default Multi-Incremental Sampling Information

Test	Subsample Basis	Aliquot	Approximate Amount per Increment	Container
Total Solids	Air Dried	15.00 g	0.50 g	2 oz. soil jar
200.7 Metals	Air Dried	1.0000 g	0.0333 g	Metals digestion tube
6010 Metals	Air Dried	1.0000 g	0.0333 g	Metals digestion tube
200.8 Metals	Air Dried	1.0000 g	0.0333 g	Metals digestion tube
6020 Metals	Air Dried	1.0000 g	0.0333 g	Metals digestion tube
Mercury	Air Dried	0.5000 g	0.0167 g	Mercury digestion cup
8081 PEST	As Received	15.00 g	0.50 g	2 or 4 oz. soil jar
8081 PEST-LL	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
8082 PCB	Air Dried	15.00 g	0.50 g	2 or 4 oz. soil jar
8082 PCB-LL	Air Dried	30.00 g	1.00 g	2 or 4 oz. soil jar
8151	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
8270	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
8270 LL	As Received	20.00 g	0.67 g	2 or 4 oz. soil jar
PAH	As Received	10.00 g	0.33 g	2 or 4 oz. soil jar
PAH ULL	As Received	20.00 g	0.67 g	2 or 4 oz. soil jar
8290/Dioxin	Air Dried	15.00 g	0.50 g	2 or 4 oz. soil jar
8330B*	As Received	10.00 g	0.33 g	2 or 4 oz. soil jar
Diesel or Residual Range Organics (DRO, RRO)**	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
TOC	Air Dried	15.00 g	0.50 g	2 or 4 oz. soil jar
Backup Sample	As Received	30.00 g	1.00 g	Back into original jar

For DOD projects refer to the DOD 8330B protocols.

** Alaska Methods AK102 and AK103 call for the extraction of from 10-30 g of sample material (soil). For MIS purposes, the minimum required amount of material per analysis is 30 g.

TABLE 2

"Large Mass" Multi-Incremental Sampling Information

Test	Subsample Basis	Aliquot	Approximate Amount per Increment	Container
Total Solids	Air Dried	15.00 g	0.50 g	2 oz. soil jar
200.7 Metals	Air Dried	10.00 g	0.333 g	Metals digestion tube
6010 Metals	Air Dried	10.00 g	0.333 g	Metals digestion tube
200.8 Metals	Air Dried	10.00 g	0.333 g	Metals digestion tube
6020 Metals	Air Dried	10.00 g	0.333 g	Metals digestion tube
Mercury	Air Dried	5.00 g	0.167 g	Mercury digestion cup or 2 oz. soil jar
8081 PEST	As Received	15.00 g	0.50 g	2 or 4 oz. soil jar
8081 PEST-LL	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
8082 PCB	Air Dried	15.00 g	0.50 g	2 or 4 oz. soil jar
8082 PCB-LL	Air Dried	30.00 g	1.00 g	2 or 4 oz. soil jar
8151	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
8270	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
8270 LL	As Received	20.00 g	0.67 g	2 or 4 oz. soil jar
PAH	As Received	10.00 g	0.33 g	2 or 4 oz. soil jar
PAH ULL	As Received	20.00 g	0.67 g	2 or 4 oz. soil jar
8290/Dioxin	Air Dried	15.00 g	0.50 g	2 or 4 oz. soil jar
8330B*	As Received	10.00 g	0.33 g	2 or 4 oz. soil jar
Diesel or Residual Range Organics (DRO, RRO)**	As Received	30.00 g	1.00 g	2 or 4 oz. soil jar
TOC	Air Dried	15.00 g	0.50 g	2 or 4 oz. soil jar
Backup Sample	As Received	30.00 g	1.00 g	Back into original jar

* For DOD projects refer to the DOD 8330B protocols.

** Alaska Methods AK102 and AK103 call for the extraction of from 10-30 g of sample material (soil). For MIS purposes, the minimum required amount of material per analysis is 30 g.

TABLE 3
Storage of Multi-Incremental Subsamples

Test	Storage
Total Solids	Room Temperature
200.7 Metals	Room Temperature
6010 Metals	Room Temperature
200.8 Metals	Room Temperature
6020 Metals	Room Temperature
Mercury	Room Temperature
8081 PEST	4 ± 2°C
8081 PEST-LL	4 ± 2°C
8082 PCB	Room Temperature
8082 PCB-LL	Room Temperature
8151	4 ± 2°C
8270	4 ± 2°C
8270 LL	4 ± 2°C
PAH	4 ± 2°C
PAH ULL	4 ± 2°C
8290/Dioxin	Room Temperature
8330B*	4 ± 2°C
Diesel or Residual Range Organics (DRO, RRO)*	4 ± 2°C
TOC	Room Temperature
Backup Sample	4 ± 2°C

FIGURE 1
Air Dried Sieve Data Benchsheet Template

ALS Inc.

Service Request Number(s):

Air Dried Sieve Data

Service Request #	Sample Weight (g)	Weight of Passing Fraction(g)	Weight of Retainied Fraction (g)	Sieve Size
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			
	0.00			

Balance ID:	
Analyst:	Date:
Reviewed:	Date:

R\:ICP\misc\digforms\Air Dried Sieve Bench Sheet-Compatible